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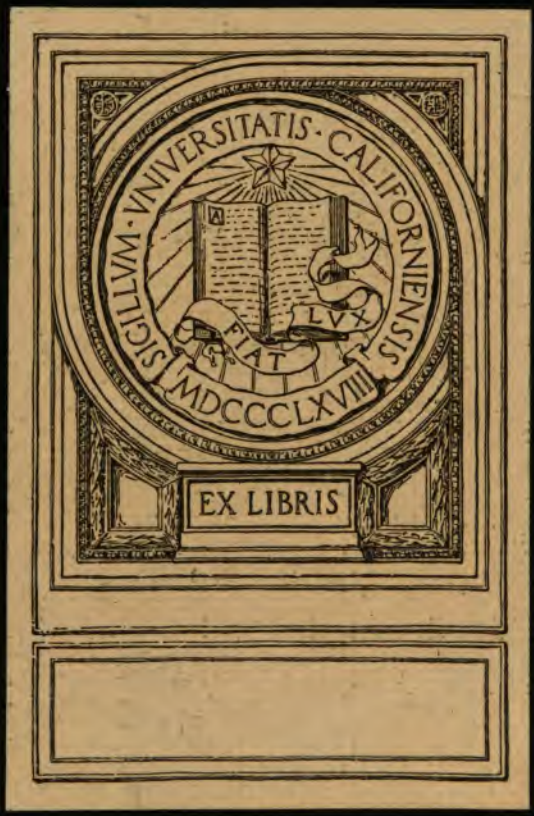
Modern Coking Practice

T. H. Byrom
and
J. E. Christopher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MODERN COKING PRACTICE

THREE BATTERIES, 132 SEMET-SOLVAY COKE OVENS, WIGAN COAL AND IRON CO., LTD.

Frontispiece.

MODERN

COKING PRACTICE

AND

CHRISTOPHER

PLATES

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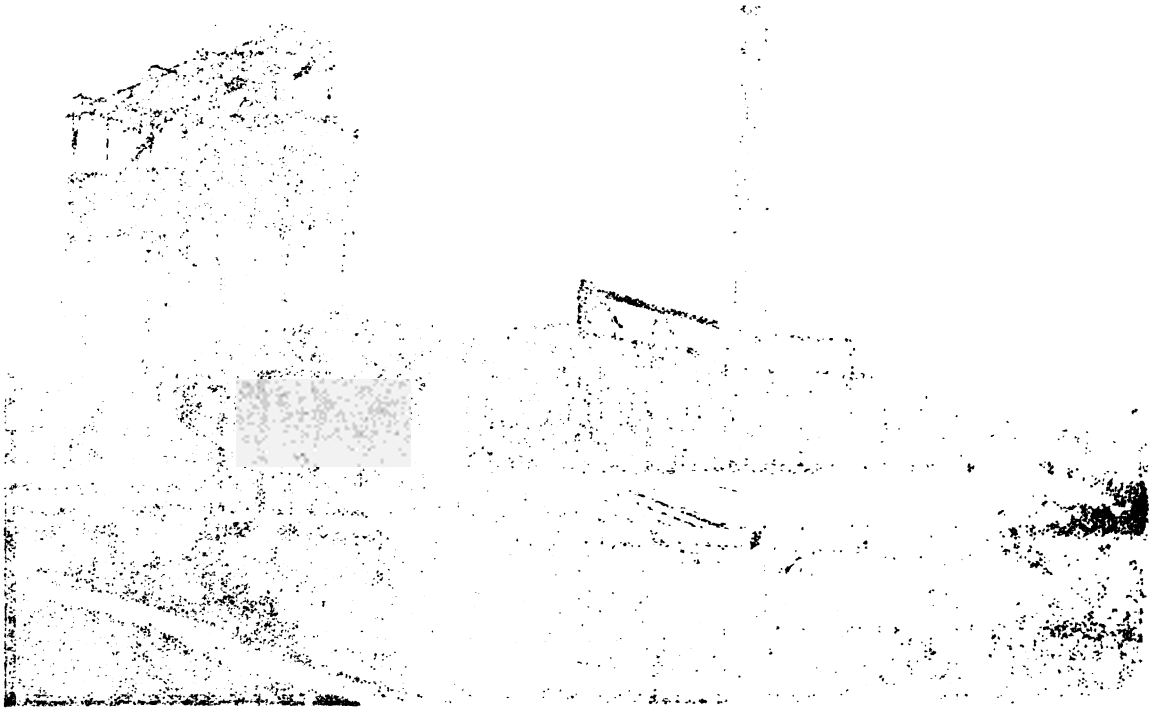

MODERN

COKING PRACTICE

INCLUDING THE

Analysis of Materials and Products

A HANDBOOK FOR THOSE ENGAGED IN COKE MANUFACTURE AND THE RECOVERY OF BYE-PRODUCTS

BY

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WITH NUMEROUS ILLUSTRATIONS AND FOLDING PLATES

NEW YORK

THE NORMAN W. HENLEY PUBLISHING CO.

132 NASSAU STREET

LONDON

CROSBY LOCKWOOD AND SON

1910

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PREFACE.

THE subject of Coke Manufacture is of rapidly increasing interest and significance, embracing as it does the recovery of valuable bye-products in which scientific control is of the first importance. It has been the aim of the authors, in compiling this book, to produce one which shall be of use and benefit to those who are associated with, or interested in, the modern developments of the industry.

Whilst the book embodies, with some amplifications, a series of lectures delivered by J. E. Christopher, at the Wigan Technical College, to a class of men engaged on coke ovens, the authors entertain the hope that the work will be appreciated by students generally, and by many who are engaged in coke manufacture, or who contemplate the laying down of bye-product plant. Every endeavour has been made to present the facts as completely up to date as possible; and if excuse be necessary for the production of such a book, it is that in proportion to the importance of the subject the literature relating thereto is very scanty, and the authors feel that they are not adding to an already overburdened subject.

The authors beg to acknowledge their obligation to Messrs W. H. Hewlett and T. M. Percy, of the Wigan Coal and Iron Co., for permission to use various information, and for opportunities of acquiring the same; also for the illustrations showing portions

302102

of the Company's Semet-Solvay coke plant. They are also indebted to Mr R. Forbes Carpenter, Chief Inspector of Alkali Works, and to Mr Herbert Porter, late District Inspector, for sundry valuable suggestions, and have had the advantage of access to the important conclusions contained in the Chief Inspector's Annual Reports, published under the Alkali Act.

They have further gratefully to acknowledge the kindness of the various firms whose ovens and appliances are described, in granting them the loan of blocks, drawings, and photographs used in illustrating the book, and to Messrs Philip Harris & Co., Birmingham, for blocks connected with scientific apparatus.

T. H. B.
J. E. C.

WIGAN, *October 1909.*

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UNIV. OF
CALIFORNIA

COKE MANUFACTURE.

CHAPTER I.

INTRODUCTORY.

Brief Summary of the History of Iron Manufacture and Allied Industries.

IRON was extracted so far back as 4000 B.C., but only in very small quantities, and in very crude condition. Up to 1619 iron was extracted in small blast furnaces with charcoal. The development of the blast furnace really commenced in 1619, when Dudley introduced coke as fuel. Owing to the ignorance of the correct methods of using coke this development was slow. The invention of the steam engine about 1781, and the introduction of the puddling process by Cort in 1784, hastened this development.

The next stage was the introduction of hot blast, in 1828, by Neilson. Huntsman's improvement in the manufacture of crucible steel in 1840, followed by Sir Henry Bessemer's great process of steel manufacture in 1855, and the open-hearth process of Sir W. Siemens in 1861, all served to hasten the march of progress. Within recent years the Siemens process has improved at a marvellous rate, and the output of iron from the blast furnaces has increased accordingly. Consequently a greater demand for coke has arisen. Coke up to recent years was made in beehive coke ovens, but keen competition has drawn the attention of experts to the enormous waste which occurred in the use of these ovens, and attempts to recover the valuable bye-products have been eminently successful. In 1869

the first battery of bye-product coke ovens was put down in England by Messrs Simon Carves; following this the first battery of Semet-Solvay ovens was laid down in 1886. Otto-Hilgenstock ovens were laid down a few years afterwards.

There are now several types of coke ovens, differing very slightly from each other, all aiming at the extraction of ammonia and tar, and producing coke equal in every respect to beehive coke.

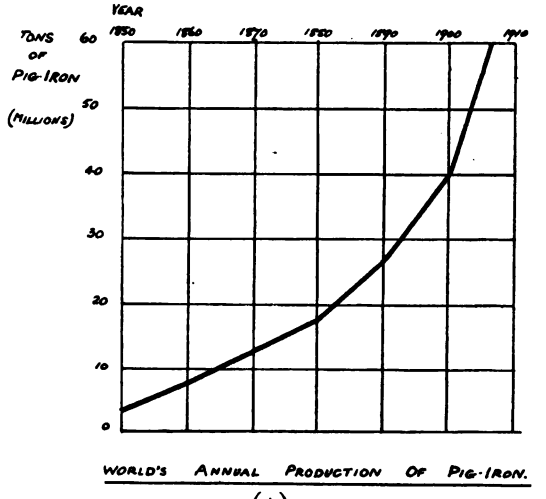
The question of bye-product extraction is an important one, and is not confined to coke ovens. Gas works, introduced about 1810, have been regular manufacturers of sulphate and chloride of ammonia, and coal-tar. Various blast furnaces, using coal as fuel, have large plants for the same purpose, and quite recently large producer gas plants have been put down with bye-product recovery plant.

	A.D.
Iron extracted from ores by means of charcoal until	1619
Coke introduced	1619
Steam engines introduced	1780
Puddling process introduced	1784
Hot blast introduced	1828
Gas engines invented	1851
Bessemer process introduced	1855
Open-hearth process introduced	1861
Coal-tar dyes invented	1863
Bye-product coke ovens introduced	1869

The following diagram (A) is given to show the enormous increase in the world's output of pig-iron since 1850. At that time the total output of pig-iron for the year was under 4½ millions. In 1906 the output was practically 60 million tons. Of this quantity the

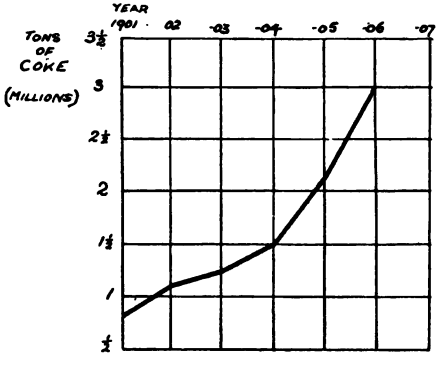
United States produced	25	millions.
Germany	12	„
Great Britain	10	„

The rapid development of bye-product coke ovens during the last few years is also shown by the following diagram (B).



WORLD'S ANNUAL PRODUCTION OF PIG-IRON.

(A)



ANNUAL COKE PRODUCTION FROM BYE PRODUCT OVENS.

(UNITED KINGDOM ONLY)

(B)

OUTPUT OF COKE FROM BYE-PRODUCT RECOVERY OVENS.

The output has thus risen from 857,850 tons in 1901 to 3,057,390 tons in 1906. The output of coke from beehive ovens is, however, very much in advance of the above. In 1906 the output from beehive ovens was about 16 million tons. This represents a loss of about 220,000 tons of sulphate of ammonia, and nearly a million tons of tar.

CHAPTER II.

General Classification of Fuels.

Practically all the operations in the manufacture of iron and steel are dependent upon heat in some form or other. Generally speaking, anything that can be practically employed for the generation of heat may be classed as fuel.

Fuels may be divided into three great classes—

Solid.
Liquid.
Gaseous.

The solid fuels may be subdivided into two classes—

Organic.
Inorganic.

Dealing with the first classification solid fuels are most largely used in iron and steel manufacture. They include—

Wood—Charcoal.
Coal—Coke.

The liquid fuels—crude petroleum, tar—are not yet extensively used in iron and steel manufacture. Still, in certain districts in Russia and America, where the oil supply is plentiful and coal comparatively scarce, naturally they are made use of. They have many advantages. Chemically speaking, oils are hydrocarbon compounds; petroleum contains from 80 to 85 per cent. of carbon combined with 10 to 15 per cent. of hydrogen, and small quantities of oxygen and nitrogen. Most oils, and especially petroleums, have a greater heating value and evaporative effect than anthracite. From the fact of their being fluid they can be burnt in the form of spray by forcing through an "atomiser," consequently they can be entirely consumed. At

the same time if care is not exercised it is possible to have much waste going on. Since oils generally contain practically no mineral matter they produce no ash and consequently no clinker; there often is, however, a sticky deposit in the flues. Usually there is little, if any, sulphur present, and therefore no corrosive sulphur dioxide is produced during combustion. As a rule the burners are so devised that they may be readily changed if desired and solid fuel used. Liquid fuel has the advantage also that its combustion may be easily and quickly started and as readily stopped, as circumstances require, without loss of fuel. The supply of a liquid combustible is evidently much more under control than that of a solid. In many cases a combination of a solid and liquid fuel is advantageous. An inferior solid fuel may be used which probably would be unfit for use alone. A combined fuel is successfully used in this country in locomotive firing. The tar, which is one of the bye-products in coking and in gas works practice, may be used as a fuel, especially if mixed off with coal or coke. The modern tendency, however, is to distil the tar first, and recover its many valuable constituents such as benzene, naphtha, carbolic acid, naphthalene, creosote, and also the tar colours discovered by Perkin. The residue consists of pitch which is usually mixed off with coal, coke, or fine slack in the form of briquettes.

Numerous patents have been taken out dealing with different methods of using tar as a fuel. Broadly speaking, the method is to either allow the tar to drip on to the solid fuel, or to feed it in the form of spray or fine jets into the combustion chamber in which solid fuel is being burnt. Arrangements are usually made whereby the tar is heated and made as fluid as possible before burning. It is said to produce no smoke when properly dealt with, and the heating power is about one and a half times that of coke.

Gaseous fuel, chiefly as producer gas, is very largely used in steel manufacture. It is made in gas "producers" though natural gas is available in some districts. The latter consists mainly of marsh gas or methane, together with some hydrogen, small amounts of other hydrocarbons, and sometimes nitrogen. Producer gas is made by passing air or a mixture of air and steam through red-hot coke. There are several types of producers, but it is scarcely necessary at present to do more than

give a few analyses of the gas from some well-known ones, as follows:—

	Siemens.	Wilson.	Morgan.
Carbon dioxide, - -	5·2	4·8	5·0
Carbon monoxide, - -	24·4	23·2	24·0
Marsh gas, - - -	2·4	1·2	3·0
Hydrogen, - - -	8·6	14·8	11·0
Nitrogen, - - -	59·4	56·1	57·0

Blast-furnace gas and coke-oven gas are now being used much more than formerly as agents of propulsion of gas engines. The great drawback has been the separation of the finely divided particles of dust with which the former is always associated to a greater or less extent. The composition of blast-furnace gas varies somewhat with the materials being smelted in the furnace, also whether coal or coke is used. An average composition from a coke-fed furnace is about:—

Carbon dioxide, - - - -	3 to 8 per cent.
Carbon monoxide, - - - -	25 to 33 „
Hydrogen, - - - - -	·3 to 7·0 „
Nitrogen, - - - - -	57 to 65 „

The nature and amount of dust in blast-furnace gas also varies with the kind of ore used and other factors.

Coke-oven gases are rich in methane or marsh gas and in hydrogen. They are used in the first place for producing the heat necessary for coking the charge in the ovens. They are also available for combustion under boilers for steam generation, for lighting purposes, and for use in gas engines. An average composition of gas from ovens of the Semet-Solvay type is as follows:—

Carbon dioxide, - - - -	1 per cent.
Olefines and Benzenes, - - -	2 to 3 „
Carbon monoxide, - - - -	1 „
Hydrogen, - - - - -	45 to 50 „
Marsh gas, - - - - -	35 „
Nitrogen, - - - - - -	4 to 8 „
Oxygen, - - - - - -	·5 „

The calorific value of such coke-oven gas averages 500 B.T.U. per cubic foot.

Organic Fuels are fuels derived directly or indirectly from substances of organic or vegetable origin.

Inorganic Fuels are of mineral and not of vegetable origin. The chief inorganic fuels are :—

Sulphur.
Silicon.
Phosphorus.
Aluminium.

Sulphur acts as a fuel in the calcination of ores containing sulphur (usually in the form of iron pyrites). In some cases there is sufficient sulphur in the ore to give quite enough heat to complete the calcination without addition of coal.

Silicon.—The action of silicon as a fuel is a very important one. The success of the Bessemer acid process of steel manufacture is entirely dependent on the heat caused by the rapid oxidation of silicon. In this process some 15 tons of molten metal containing 2 per cent. silicon is run into a "converter." Air is blown through the molten metal to oxidise the silicon and other impurities. The silicon becomes oxidised to silica, and combining with other oxides passes into the slag in the form of silicates. No external heat is applied, and yet after twenty minutes' blowing the temperature is much higher. The heat caused by this oxidation is equivalent to that given off by 8 cwt. of coal, burning away completely in twenty minutes.

Phosphorus acts in a similar manner in the basic Bessemer process. Pig-iron for this process should contain from 2 to 3 per cent. of phosphorus. The phosphorus becomes oxidised, and is taken up by lime and other basic materials present forming slag.

Aluminium is now actually used as a fuel for special purposes. The heat which can be generated is quite equal to that produced in electric furnaces. Aluminium has a very strong affinity for oxygen, and if brought into contact with metallic oxides takes up their oxygen, leaving the metal in an almost pure state. If oxide of iron be used, the iron produced

reaches such a temperature that, if poured into blow-holes in castings, &c., it actually fuses the surface of the casting, becoming practically welded to it. For this purpose the aluminium must be in a fine, granular state of division, and must be mixed in definite proportion to the oxide, according to the formulæ:—

$$3FeO + 2Al = 3Fe + Al_2O_3,$$
$$Fe_2O_3 + 2Al = 2Fe + Al_2O_3,$$
$$3Fe_3O_4 + 8Al = 9Fe + 4Al_2O_3.$$

This mixture is placed in crucible, covered with a small proportion of a priming mixture of aluminium and barium peroxide in the following proportion:—

$$3BaO_2 + 4Al = 3Ba + 2Al_2O_3.$$

This priming mixture, on being ignited by means of magnesium ribbon, brings about a local heat sufficient to start the main reaction. Other metals having very high melting points, and whose oxides are very refractory, may be reduced by the above process, such as chromium, tungsten, manganese, &c.

Organic Fuels:—

Wood—Charcoal.
Peat.
Lignite.
Bituminous coals { Coal gas. Producer gas. Coke, &c.
Anthracite.

Approximate composition of above:—

	Carbon.	Hydrogen.	Oxygen.	Nitrogen.
Wood - - - -	50	6	42	1
Peat - - - -	60	6	30	1·2
Lignite - - - -	70	6	20	1·2
Cannel - - - -	84	5·8	8	1·2
Bituminous coal - -	88	5	5	1·2
Anthracite - - -	92	3·5	2	1·2

Wood is not used to any great extent as fuel in iron and steel manufacture. It contains, even when well seasoned, a

large proportion of moisture (18 to 20 per cent.), which reduces the heat effect very largely.

Charcoal.—When wood is converted to charcoal, the moisture and other volatile substances are expelled, leaving a material consisting practically of carbon only. Charcoal bears the same relation to wood as coke does to coal.

Peat.—This is not used as a fuel in iron and steel manufacture. It is practically an accumulation of vegetable matter, varying in composition according to the age and depth of deposit. One great objection to its use is the high percentage of ash and its power of retaining moisture.

Lignite, or brown coal, though used on the Continent, is rarely used in this country. It may be looked upon as a link between peat and the bituminous coals. It is usually contaminated with earthy matter.

Bituminous Coals are black in colour, and vary slightly in composition and physical properties. They may be divided into two great classes:—

1. Caking.
2. Non-caking.

All coals, caking or non-caking, consist of:—

Fixed carbon	-	Coke.
Volatile matter		Marsh gas, carburetted hydrogens, hydrogen, tar, &c.
Ash - - -		Incombustible matter, silica, oxide of iron, &c.

On subjecting coals to heat (out of contact with air), the volatile matter is expelled as coal gas, leaving the fixed carbon in the form of coke. This coke retains the earthy or mineral matter of the coal. If the coke be burnt with a full supply of air, it is converted to carbon dioxide, and the mineral matter is left in the form of ash.

Impurities in coals:—

Ash.
Sulphur.

Ash:—

ANALYSIS OF COAL ASH.

Silica	30 to 60 per cent.
Alumina and Oxide of iron	35 to 55 „
Lime	5 to 10 „
Magnesia	traces to 2 „
Sulphuric acid	3 to 8 „
Phosphoric acid	·5 to 1·5 „
Potash and Soda	2 to 4 „

From the above it will be seen that the ash is composed chiefly of silica and oxide of iron. In blast furnaces this silica would have to be fluxed away with limestone, thus increasing the working charges of the furnace. The colour of the ash is a very reliable indicator of the amount of oxide of iron in the ash, oxide of iron having a reddish-brown colour. On burning a coal, any pyrites or sulphide of iron becomes converted into oxide, so a reddish-brown ash is also an indicator of sulphur in the coal.

Sulphur.—The sulphur, which is a very objectionable constituent in fuels generally, exists in coal in four conditions:—

1. As iron pyrites (coal brasses) (iron bisulphide).
2. As sulphate of lime, and sometimes as sulphate of alumina.
3. In an organic form, combined with carbon and hydrogen.
4. In rare instances as free sulphur.

Of these, that which occurs in the form of pyrites is the most injurious, because, on combustion of the fuel, its sulphur becomes oxidised to sulphur dioxide, which in presence of moisture becomes sulphurous acid, and eventually, by further oxidation, sulphuric acid. This has an intensely corrosive effect on any iron or steel with which it may come in contact, especially the cooled surfaces of economiser tubes, or any other parts on which moisture can condense, thus absorbing more readily the acid fumes. The sulphur in the form of sulphate is not injurious at all, except that it adds to the mineral constituents or ash of the coal. Iron pyrites in coal is also objectionable in this manner. On burning the coal, the oxide of iron formed becomes reduced to ferrous oxide, and then combines with the silica and alumina of the ash, forming a fusible mass known as "clinker." This substance is very

objectionable, not only because it is waste material, but because it clogs up the fire-bars, preventing a free passage of the air required for combustion, and carries away with it unconsumed carbon in the form of cinders.

Coke is made by carbonising coal in suitable ovens, where it is heated out of contact with air, or with the admission of as little air as possible. The old type of beehive oven is being rapidly displaced by more modern forms, such as the Simon Carves, Semet-Solvay, &c., the principles of which are explained fully in describing these various ovens. They differ materially in many ways from the beehive form, the coal being coked in chambers by hot gases which circulate outside them, the gases being produced from the coal itself.

The object of coking is to drive off volatile matter, which increases the percentage of fixed carbon in the residue, also because slack can be used which in many cases would be quite useless for metallurgical and foundry work without carbonising. A portion of the sulphur existing as pyrites in the coal is got rid of during coking, as well as a portion of that existing in the organic form. Some also is expelled during quenching, owing to the action of the hot sulphides on steam, producing sulphuretted hydrogen.

Coke is better in some respects than coal for steam raising purposes, given a good draught of air. It is not liable to spontaneous combustion, and requires more heat to set it alight. The yield of coke from any particular slack as obtained in a laboratory test is usually somewhat higher than that obtained by actual results in the ovens. This is partially due to the unavoidable admission of air with the charge. On the other hand, this is counterbalanced to some extent by the decomposition of some of the hydrocarbons which are driven off, resulting in the deposit of solid carbon. This may be often seen in the form of thread-like masses in the coke, sometimes also in small rounded masses of very dense material with a bright metallic lustre, chiefly composed of carbon, and practically free from ash. All the mineral matter originally in the coal remains in the coke, and consequently the percentage in the coke is proportionally greater.

There was considerable prejudice for some time against the use of coke made in a plant from which bye-products were also

recovered, and possibly this was well founded in the early days of bye-product development. It was generally accepted then that the simultaneous production of good coke and the recovery of marketable bye-products was not possible. That prejudice has, however, been forced to give way to the undoubted fact that the best coke can be made by modern processes, with the accompanying benefit of ammonia, tar, and light oils recovery.

Coke contains, in addition to fixed carbon and ash, variable amounts of oxygen, hydrogen, and nitrogen. Most probably the two former are occluded, but there is always more or less nitrogen present, however high the temperature employed in carbonising. In the blast furnace, coke plays a two-fold part; firstly, as a source of heat to maintain the temperature necessary for the various chemical reactions; and secondly, to act as a reducing agent by the production of carbonic oxide. In foundry work, this latter reaction is not requisite in the cupola, in fact the aim ought to be to burn the coke completely to carbonic acid (carbon dioxide), and thus get its full heating effect. Coke for use in a blast furnace must be hard and dense, so as to be able to resist the crushing force of the weight of materials in the furnace. A soft, friable coke leads to the accumulation of masses in the furnace, which leads to trouble by causing "scaffolding" or "hanging." Soft coke also produces more dust, which passes away with the gases escaping from the furnace, and dusty gas is one of the problems which the modern blast furnace manager has to cope with in view of the extending use of waste gas in gas engines. It is well known that some coals will not coke under any conditions, while others are highly suitable for coke making. Between the two extremes are many qualities from which varying grades of coke may be made.

The Coking Power of Coal.—This may be determined by heating a portion with varying amounts of sand. The more sand a coal can be mixed with, and still retain its coking power, the better its coking quality. The number of grams of sand per gram of coal forms a standard of comparison. At the bottom of such a scale we should find the non-caking coals, such as anthracite, while a strong caking coal would give as high a figure as 14 or 15.

As sulphur is such an objectionable element in any fuel

which is to be used in a blast furnace producing iron, many devices have been tried to eliminate as much as possible from the coke itself. Quenching with various solutions has repeatedly been tried, but as a rule with very moderate success. Generally speaking, little can be done in this way, and especially so when the ash contains much iron, and also lime and magnesia, since these retain sulphur most strongly. The most satisfactory method is to clean the *coal* as far as possible before coking.

CHAPTER III.

Coal Washing.

Coal Washing.—Coal, as it comes from the mine, is always contaminated to a greater or less degree with impurities of various kinds which, though practically unavoidable, lessen considerably the value of the coal for most purposes. Some of these, such as silicious and calcareous shaley matter, and pyrites, are actually associated with the coal itself, whilst others get into the coal from the roof and floor during the operation of mining. It is obviously of material benefit, both to seller and consumer, if these foreign substances can be removed in some way, and the method usually adopted is to treat the coal in some form of washing apparatus whereby it is rendered more or less free from the above-mentioned materials. There are several types of washers in general use, and it will be the purpose of this chapter to describe a few of the washers used in this country.

There are many points to be dealt with in considering the washing of coal, given an effectual type of washer. There is the cost in the first instance, then the physical nature and condition of the coal itself, some coals being much more friable than others, producing more "smalls" in the mining, then the specific gravity of the coal and impurities, shale, pyrites, &c. Again there may be several kinds, such as bone coal, cannel, and shaley coal, in the same seam, and the specific gravity of these will probably differ appreciably from that of the main bulk. The great point to be aimed at is, of course, to wash the coal as completely as possible, removing a maximum amount of impurity, with a minimum amount of coal passing with the dirt. The analysis of representative samples of the material before and after washing will give the percentage elimination of mineral matter and sulphur. It must be remembered that

even the cleanest piece of coal which can possibly be selected is by no means absolutely free from ash, in fact, some coals, picked as clean as possible, will contain 10 per cent. or more of mineral matter. Now no amount of washing can possibly remove such substance because it is ingrained in the substance of the coal itself, and no doubt got there during the formation of the seam, ages ago. In fact much of it is due to the ashes of the plants from which the coal was formed. The amount of coal passing away with the dirt should be carefully watched, and this may be readily done by taking the specific gravity of the coal, and of the dirt separated from it. For the moment assume that the specific gravity of the coal is 1·25 and that of the shale 2·5 and pyrites 3·5. It is evident that by making a solution of

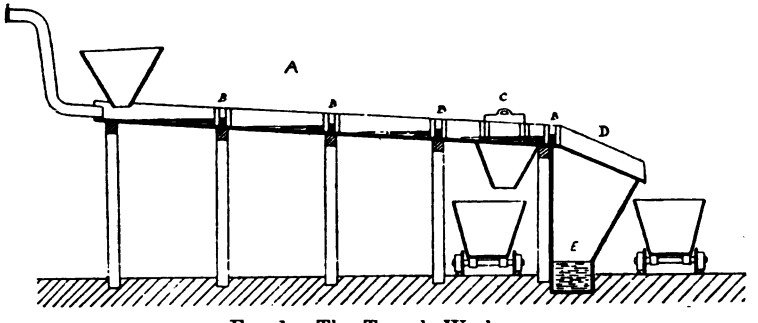


FIG. 1.—The Trough Washer.

calcium chloride (or other suitable substance) with a specific gravity of 1·3 to 1·35 we shall have a liquid in which shale and pyrites will sink and coal will float.

Hence, if a weighed portion of the dirt be taken (500 or 1,000 grams), placed in the above solution, and well stirred up, the coal which floats can easily be skimmed off, placed in a filter paper, washed free from calcium chloride, dried and weighed, giving the percentage of coal in the dirt by a simple calculation.

There are various types of coal washers, and in all of them the coal is agitated in a good supply of water, the agitation being brought about by various means, viz.:—

(*a*) The flow of the water itself.
(*b*) Travelling belts and scrapers.
(*c*) Revolving arms.
(*d*) Pulsation of the water by pistons or compressed air.

The simplest and most elementary type of coal washer is the trough washer (Fig. 1). It consists of a trough A varying in length from 40 to 100 feet, about 3 feet in width and about 15 inches deep. At intervals movable dams B are placed. The

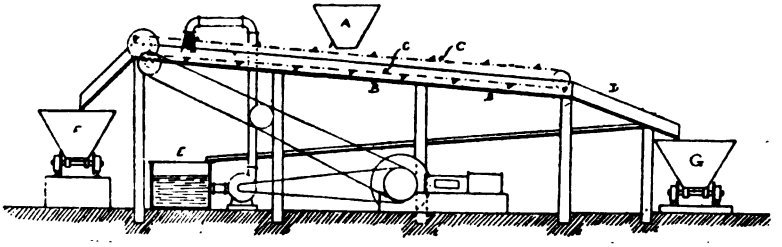


FIG. 2.—The Elliott Washer.

trough is set at an inclination sufficient to cause the necessary agitation of the coal for the efficient settling of the heavier shale and pyrites. This dirt is caught by the dams B and at

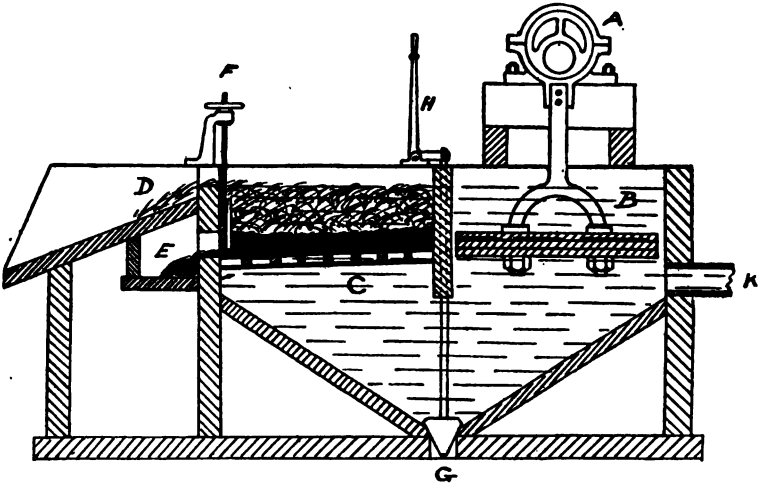


FIG. 3.—The Jig Washer.

intervals is run off from the side of the trough through the sluice C. The washed coal passes over a screen D, the water passing through into the channel E.

In the Elliott washer (Fig. 2) the principle of trough washing is taken advantage of, but the trough is made more efficient by

using a scraper chain to agitate the coal more effectively. By careful regulation of the inclination of the trough and of the speed of the chain a very effective separation can be brought about. The main points of this washer can easily be understood from the sketch on page 16.

Another type of coal washer is the "Jig" washer (Fig. 3). There are various modifications, but the sketch on page 16 will explain the principle. The "jig" is divided into two portions. In one of these an oscillating piston B moved by an eccentric or crank arrangement serves to agitate the water, supplied either by the pipe K or along with the coal. In the other portion is a grid C supporting a perforated copper screen. The coal is fed on to this grid and the "throbbing" action of the water causes the dirt to settle on this bed. The fine dirt passes

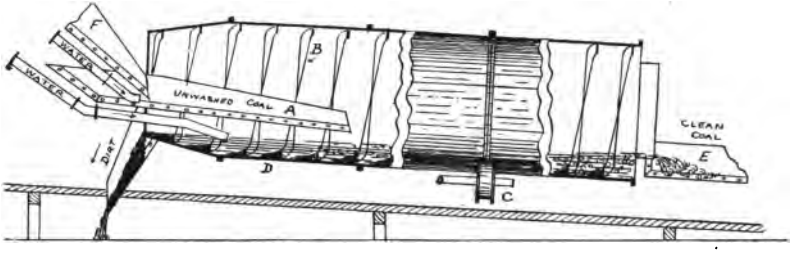


FIG. 4.—The Blackett Washer.

through and is collected at intervals by lifting the plug G. The rough dirt is run off occasionally by raising the sluice F. For finer slacks a bed of felspar is supported on the grid C. The dirt being heavier than this felspar, sinks through it. The washed coal being lighter remains above the felspar and is washed over a screen D into elevators or trucks, the water passing through the screen being pumped back into the "jig." The length of stroke is adjusted according to the size of coal treated, thus 1 inch to 1½ inch nuts require a stroke of about 3 inches, whilst coal of about ¼ inch size would only require a stroke of about 2 inches. The revolutions of the jig also increase as the size of coal decreases, the 1½ nuts as above requiring about 90 revolutions per minute, and the ¼ inch coal about 120 revolutions per minute.

The Blackett washer (Fig. 4) is a type of washer in which

the agitation is brought about by means of a revolving barrel, set at an inclination to suit the size of coal washed, &c. At the same time a shallow worm attached to the inside of the barrel gradually forces the heavier "dirt" to the upper end of the barrel, where it is discharged, whilst the true coal is washed over the worm and delivered at the lower end on to a drainer.

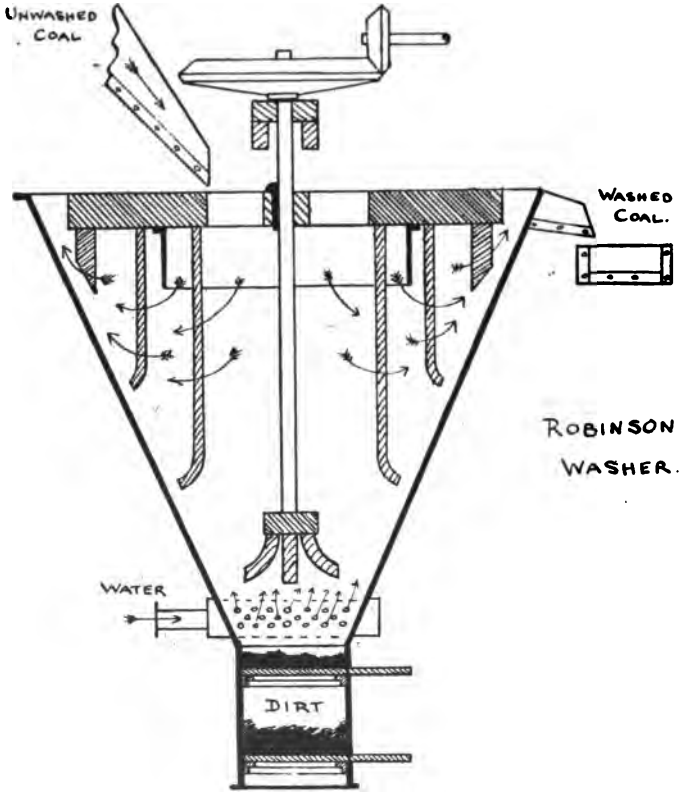


FIG. 5.—The Robinson Washer.

The Robinson washer (Fig. 5) consists of a funnel-shaped chamber, the contents of which are kept in motion by means of revolving arms. The water is pumped in at the bottom of the cone and flows over the edge of the funnel, carrying the washed coal with it. The dirt settles to the bottom, and is removed by means of the double-doored arrangement shown in sketch.

The Baum washer (Fig. 6) shown below is a very effective type. The agitation is brought about by means of compressed air. The removal of the dirt will be readily understood from

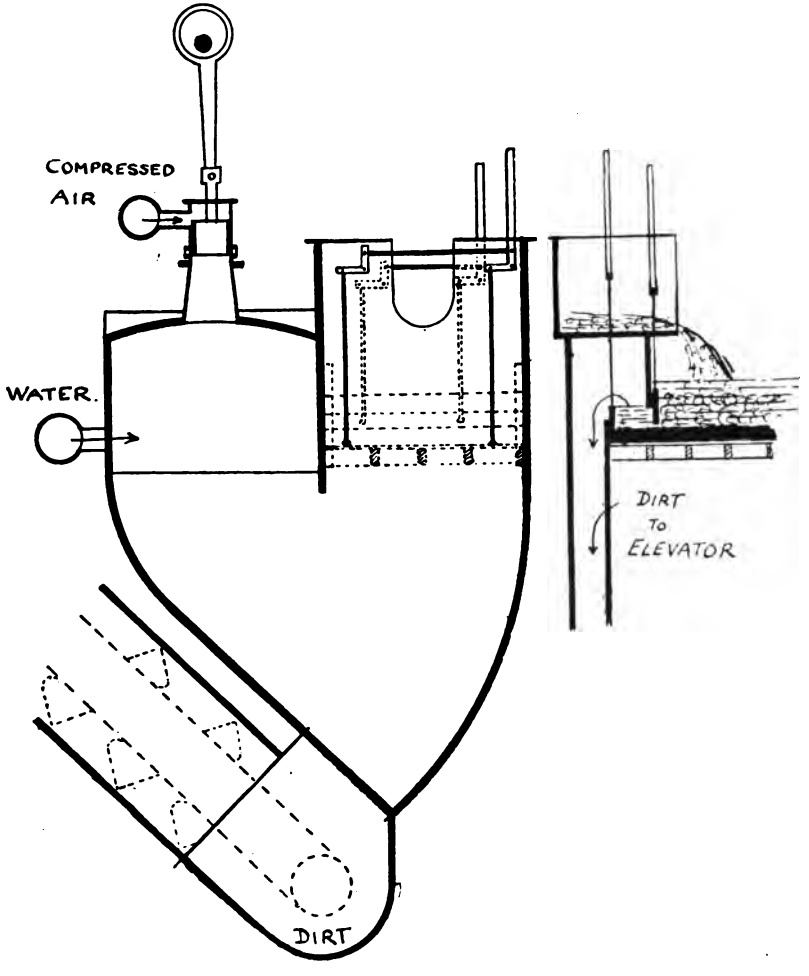


FIG. 6.—The Baum Washer.

the drawing. The use of compressed air makes the pulsation more elastic. There are fewer working parts, and the wear and tear is consequently very much reduced.

CHAPTER IV.

Sampling and Valuation of Coal—Nitrogen in Coal.

Sampling and Valuation of Coal, Coke, &c.—In making a physical or chemical examination of coal or other material, it is obviously of first importance that a representative sample of the substance be obtained. It is impossible for one or two small pieces, say of coal, to represent fairly an average composition of any particular seam; they may be either too clean (which is the more likely) or too shaley. At the least, several barrows full of coal should be taken and roughly broken down, in the first instance, on a clean surface, preferably iron or steel. The heap should then be well turned over and mixed up, spread out, and divided crosswise into quadrants. Opposite sections should then be taken away, thus halving the total sample. The remainder is crushed smaller, again thoroughly mixed up and quartered, rejecting half. This is repeated until the sample has been reduced to a reasonable quantity, say 10 or 12 lbs., which should then be crushed, if necessary, until all passes through a sieve of about $\frac{3}{16}$ inch mesh. From this a smaller sample is taken after thorough mixing, crushed somewhat smaller, and a final sample of 1 or 2 oz. crushed so as to pass an 80 sieve. It is then ready for analysis, and is a fair average sample of the material.

Specific Gravity of Coal, Coke, Shales, &c.—The specific gravity of solids insoluble in water, such as coal, shale, pyrites, &c., may be readily determined, given a moderately accurate balance and set of weights. The test may be done on small lumps or on the powdered material. If lumps are used, then several determinations are desirable on different pieces, taking the average result as the true figure. If the powdered substance is used, only one, or at the most two determinations will be

needful. In these determinations the principle of Archimedes is made use of, namely, that when a body is submerged in a liquid it loses weight equal to the weight of liquid displaced by it. In an actual determination, if a small lump is being used, it is suspended by a loop of horsehair from one end of a balance and carefully weighed. It is then immersed in water in a glass vessel, supported on a wooden bridge (Fig. 7), and again weighed.

A considerable decrease in weight will be noticed, due to the operation of the above-named principle. If W be its weight in

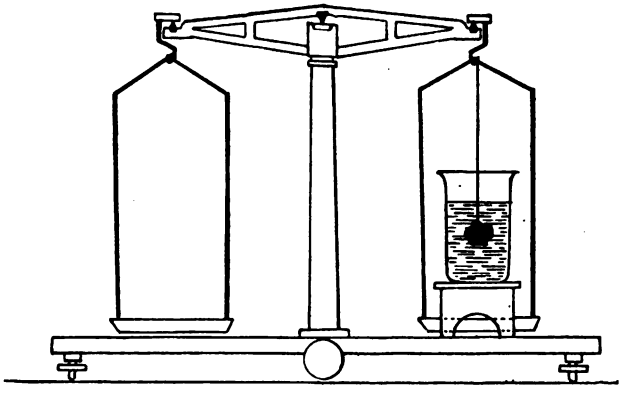


FIG. 7.—Balance, &c., for Specific Gravity Determination.

air, and w its weight in water, then the specific gravity of the solid is:—

$$\frac{W}{W-w}$$

Example:—

Weight of sample in air - - -	16·342 grams.
Weight of sample in water - -	4·098 „
Weight of water displaced - -	12·244 grams.

$$\text{Specific gravity} = \frac{16{\cdot}342}{16{\cdot}342-4{\cdot}098} = 1{\cdot}334.$$

In the case of a powdered sample, it is, of course, impossible to use this method, and the use of a specific gravity bottle is necessary. This is a small bottle (Fig. 8), fitted with an accur-

FIG. 8.—Specific Gravity Bottle.

ately ground stopper, through which a fine hole is drilled lengthwise, to enable the bottle to be completely filled with water or liquid, which is not easily done with the usual solid stopper. It is first carefully filled with distilled water and the stopper inserted. After carefully wiping off all superfluous liquid from the outside, the bottle is weighed. A portion of the water is then emptied out, and 1 gram of the powdered coal or other mineral placed carefully in the bottle, and well shaken to thoroughly wet the material. This is not at all easy in the case of coal, and great care should be taken that all air bubbles, which are readily enclosed by coal dust, are completely got rid of, otherwise the result will be inaccurate. Finally, fill up with water, replace stopper and weigh. An example will best illustrate the calculation of the result:—

Weight of specific gravity bottle filled with water - - - - - -	73·498 grams.
Weight of bottle containing water and 1 gram coal - - - - - -	73·745 „
i.e., weight of bottle and water left in after displacement by the coal - - - - - -	72·745 „

∴ Weight of water displaced
$= 73{\cdot}498 - 72{\cdot}745$ or ·753 gram.

∴ Specific gravity $= \frac{1}{{\cdot}753}$ or 1·328.

The specific gravity of liquids may be obtained either by use of some form of hydrometer or by means of the specific gravity bottle. The Twaddell hydrometer (Fig. 9) is in common use in this country. The readings obtained by its use are readily converted into specific gravity by multiplying by 5 and adding 1,000 (taking the specific gravity of water as 1,000). Thus:—

FIG. 9. Twaddell Hydrometer.

If the hydrometer floats at 65°, then taking specific gravity of water as 1,000, the specific gravity of sample would be $(65 \times 5) + 1{,}000$, or

1,325; or taking specific gravity of water as 1, the specific gravity of the sample would be 1·325.

Conversely, if the specific gravity is given to three decimal places, neglect the decimal point, subtract 1,000 and divide by 5. The result will indicate degrees on the Twaddell scale. A saturated solution of calcium chloride at a temperature of 15° Cent. contains 40·66 per cent. of the salt, and has a density of 82·2° Twaddell, which is equal to a specific gravity of 1·411. If the specific gravity bottle be used, it is first carefully weighed empty and dry, then filled with distilled water at about 15° Cent. and weighed again. The water is emptied out and replaced by the liquid whose specific gravity is required (the temperature being the same as that of the water), carefully filled and weighed. The results give the relative weights of equal volumes of water and the liquid to be tested. From this the specific gravity is readily obtained. Thus:—

Weight of bottle filled with water - -	45·438 grams.
Weight of bottle (empty and dried) -	20·652 „
Weight of water - - - - - -	24·786 grams.
Weight of bottle filled with liquid - -	50·672 grams.
Weight of bottle (empty and dried) -	20·652 „
Weight of liquid - - - - - -	30·020 grams.

Hence the specific gravity of the liquid is:—

$$\frac{30{\cdot}020}{24{\cdot}786} \text{ or } 1{\cdot}211.$$

It is often important to determine the amount of moisture in coal, as even that which is freshly mined contains appreciable amounts, usually from 2 to 5 per cent. If the mineral has been exposed to wet weather, it is very likely to contain more. Some coals will take up and retain more moisture than others, owing to their physical condition. To ascertain the amount, a fair average sample is taken and crushed down to not less than ¼ inch size, a weighed quantity taken (say 100 or 250 grams), and dried for three or four hours at a temperature of 100° Cent. in a water bath, allowed to cool, and the loss ascertained by weighing.

If the sample be reduced to a very fine condition, it is liable

to lose other constituents than moisture on prolonged heating, even at 100° Cent. Also pyrites may become oxidised. A safer plan, and one which is useful as a check, is to expose a weighed portion under a bell-jar desiccator for at least twelve hours, and weigh the loss due to moisture, which is taken up by the sulphuric acid or calcium chloride used in the desiccator. In this way there is not the same danger of driving off easily volatile substances along with the moisture.

Estimation of Ash in Coal and Coke.—The amount of ash contained in coal or coke may be determined by taking a weighed portion from the finely powdered sample and igniting it carefully, either over the straight Bunsen flame or preferably in a gas muffle, cautiously and gradually at first to avoid loss by decrepitation, increasing the heat until finally a good red heat is reached, combined with a plentiful air supply. It will be found that coal burns off more quickly than coke. In the case of hard burnt coke the ash should be weighed until it ceases to lose weight.

When a coal is completely burnt off it ceases to glow, but it is not easy to judge a coke in this way, and especially if the ash is high, as this protects the small particles of hard coke from oxidation, so that occasional careful stirring is desirable. Small porcelain crucible lids, about $1\frac{3}{4}$ inches in diameter, are very suitable for burning off coke or coal. It will be found that if platinum vessels are used, the metal becomes brittle and cracks after a time, probably due to prolonged heating in contact with carbon. After all the carbonaceous matter has been oxidised, the ash is carefully brushed out of the dishes, when cool, on to the balance pan and weighed. Thus, if 1 gram of coal gave an ash weighing ·087 gram, percentage of ash in the coal would be $100 \times \cdot 087$, or 8·7 per cent.

The colour of the ash should be noted. Since all the mineral matter which is in the original coal, with the exception of some of the sulphur, remains in the coke, it is obvious that the ash in the coke is higher than that in the coal from which it is made. For instance, if a coal contains 7 per cent. of ash, and yields 70 per cent. of coke, the ash in the latter will be $\frac{7}{70} \times \frac{100}{1}$ or 10 per cent.

Estimation of Volatile Matter.—The volatile matter in coal is that portion which is driven off or volatilised, when the fuel is heated out of contact with air. It usually includes some portion of the sulphur which is present in the coal, and it is generally taken that about 50 per cent. of the sulphur is thus driven off. The volatile matter is estimated by heating a weighed portion in a closed crucible, without admission of air. One or two grams may be taken and gently heated over a Bunsen flame until smoke ceases to issue from the crucible; it is then quickly transferred to a muffle furnace at a good red heat, the furnace door closed and the heating continued for two and a half or three minutes, after which the crucible and contents are removed, placed under a bell-jar desiccator till cool, and then weighed. The loss represents volatile matter, and the residue, coke, *i.e.*, fixed carbon plus ash or mineral matter. Theoretically coke ought not to contain any volatile matter, but usually a slight amount is present, say from ·5 to ·8 per cent. The volatile matter in coals varies with the kind of coal, as may be seen from analysis (page 151).

Estimation of Sulphur.—The sulphur in coal and coke may be estimated by

(*a*) the Lime method, or
(*b*) Eschka's method.

(*a*) Weigh off carefully 1 gram of the fuel and 1 gram of pure lime, on which a blank estimation for sulphur has been done. Moisten to a fairly stiff paste with distilled water and mix up intimately with a spatula or glass rod, being careful to avoid any loss. Dry slowly in a water bath or under a hot plate, and place in front of the muffle furnace for about half an hour, giving free access of air. Afterwards heat strongly in the muffle for a further half hour. By this treatment the sulphur in the fuel becomes converted into sulphate of lime. After removal from the furnace and cooling, the mass is transferred completely to a small beaker, and moistened with distilled water. Sufficient hydrochloric acid, containing a few drops of bromine (both free from sulphur), is added to dissolve the calcium sulphate and excess of lime. The solution is then boiled and filtered. After washing the filter paper, the

filtrate is boiled, and while boiling a hot solution of barium chloride is added cautiously. This reacts with the calcium sulphate in solution, forming barium sulphate, which is precipitated as a heavy white powder.

It should be allowed to settle completely, which will probably take a few hours, then filtered off through Swedish paper, taking care that none passes through the pores of the filter. It is finally washed, dried, ignited, and weighed as barium sulphate (which contains 13·73 per cent. of sulphur). From this the percentage of sulphur in the fuel may be readily obtained. Thus 1 gram of coal yielding a precipitate of barium sulphate weighing ·12 gram would contain ·12 × 13·73 grams of sulphur, equivalent to 1·647 per cent.

(*b*) In Eschka's method 1 gram of the fuel is taken, finely divided, and mixed in a shallow dish with about 1 gram of pure calcined magnesia and about ½ gram anhydrous sodium carbonate. The mixture is then covered with a thin layer of magnesia (about ½ gram). As in the case of the lime method a blank determination of the sulphur in the material used must be made, and the weight of barium sulphate obtained (if any) deducted from the total weight finally obtained. The dish and contents are placed in front of the muffle furnace for about three-quarters of an hour, allowing free access of air, then finally heated somewhat more strongly for fifteen or twenty minutes. After removal from the muffle and cooling, the contents of the dish are carefully transferred to a beaker and hot distilled water added, with a few drops of bromine and hydrochloric acid, boiled and filtered clear of insoluble residue. After washing, the solution obtained is boiled up and barium chloride added, as in the lime method, to form a precipitate of barium sulphate. The subsequent operations are carried out as in method (*a*). In this method the sulphur of the fuel becomes converted into the sulphates of magnesium and sodium, both of which are soluble in water. The bromine oxidises any sulphides that may have been formed to sulphates as in (*a*).

When coal is heated in a closed oven or retort, as in the various methods of coking, it is said to be subjected to destructive distillation. It is broken up into a number of products, solid, liquid, and gaseous, the proportion of which largely depends upon the temperature employed and the nature of the particular

coal which is being carbonised. If the temperature is high the products are mainly gaseous. On the other hand, if the temperature is low, heavy tarry liquids are formed, which retain various solid substances either in solution or suspension. Under normal conditions of coking the volatile hydrocarbons are almost completely expelled. A well-burnt coke ought not to contain more than 1 per cent. of volatile matter. Along with the volatile substances are other gaseous products, such as cyanogen, ammonia, carbon dioxide, sulphuretted hydrogen, and free nitrogen.

All coals contain more or less nitrogen, varying from 1 to 2·5 per cent. On heating, a portion of this nitrogen is driven off in the free condition, while some is retained by the coke. Some of the liberated nitrogen combines with the hydrogen to form ammonia, which constitutes a valuable bye-product eventually. Another portion continues, with carbon, to form cyanogen compounds, and some of the nitrogen remains in the free condition, going into the gases as such. It has been estimated that only about 12 to 14 per cent. of the nitrogen originally present in the coal goes to form ammonia.

The amounts of ammonia and cyanogen formed in coking depend on several conditions, one of which is the physical condition of the coal—that is, with regard to lumps and small. If small and compact, the mass takes longer to heat up, and consequently the products are different than when the mass is heated up more quickly. Then, again, the air spaces in a mass of coarse material have an important bearing on the composition of the gaseous products. The dampness or dryness of the coal is important. In the former case there is usually more ammonia and tar, also sulphuretted hydrogen and carbon dioxide. If the temperature of coking is high, it results in the formation of more cyanogen compounds.

The question of temperature with regard to ammonia yield is undoubtedly most important, because if too high a temperature is being employed the ammonia becomes decomposed again into nitrogen and hydrogen. It is estimated that at about 800° Cent. the maximum amount of ammonia is obtained. Consequently it follows that the longer the contact of the gases with the heated retort surfaces the more likelihood of a low yield of ammonia, especially so if high temperatures are used for carbon-

ising. The composition of the ash of the coal, no doubt, has an effect also on the ammonia yield.

M'Leod, in a paper read before the Society of Chemical Industry in 1907, from a consideration of many analyses, allots the distribution of the nitrogen as follows:—

Nitrogen in the coke - - -	58·3	per cent.
„ „ tar - - -	3·9	„
„ „ ammonia liquor	17·1	„
„ as cyanogen - - -	1·2	„
„ in the gas - - -	19·5	„

The cyanogen compounds are in many cases, in modern coking and gas works practice, largely recovered in some form or other, such as sodium cyanide, Prussian blue, sodium ferrocyanide, &c. In these forms they constitute saleable bye-products, at the same time minimising the danger to workmen which arises from the presence of hydrocyanic acid in waste and other gases.

The waste gases coming from the saturators of a sulphate plant often contain as much as 2 per cent. by volume of hydrocyanic acid, and about the same quantity of sulphuretted hydrogen. The latter may be conveniently and easily dealt with in a Claus plant for the recovery of sulphur in the solid form; at the same time a portion of the cyanogen becomes converted into ammonia. The Claus plant is more fully described later (page 115).

The Estimation of Nitrogen in Coal and Coke.—The method most suitable is that known as the Kjeldahl process, which consists in heating the finely divided material with concentrated sulphuric acid in presence of manganese dioxide. The effect is to destroy all the carbonaceous and organic matter, and at the same time to convert the whole of the nitrogen into ammonium sulphate. On then adding a moderate excess of sodium hydrate solution and distilling into a standard solution of sulphuric acid, the amount of ammonia liberated is calculated from the quantity of sulphuric acid neutralised.

As to quantities, 1 gram of coal is a suitable amount to use. Heat with 25 to 30 cubic centimetres of strong sulphuric acid for half an hour. Then add 5 grams of manganese dioxide

and allow to boil forty-five minutes, and a further 5 grams, heating finally for another forty-five minutes.

Allow the mass to cool, and *very carefully* add a little water. Rinse the whole out into a copper flask (glass vessels are useless), adding a solution of sodium hydrate containing about 25 grams of the solid. Distil into a solution of normal sulphuric acid; about 15 c.c. diluted is usually sufficient. Each 1 c.c. of acid neutralised represents ·014 gram of nitrogen. It is advisable to make a blank determination on the materials used.

CHAPTER V.

Calorific Power of Coal and Coke.

Calorific Power.—The calorific power or heating value of a fuel, and hence its evaporative power, can be determined by several methods, in broad principles the same, namely, the combustion of the fuel in oxygen (supplied either in the solid or gaseous form), and the measurement of the heat thus produced. It is possible, also, to calculate, from an elementary analysis of the fuel, the calorific power. The results, however, do not agree very closely with those found in a practical test.

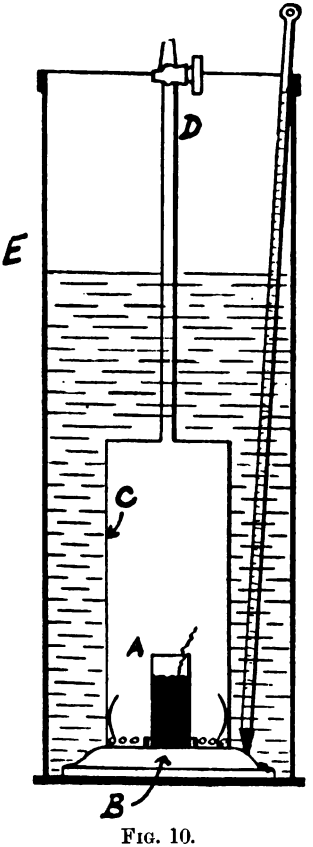


Fig. 10.
Lewis Thompson Calorimeter.

In the Lewis Thompson calorimeter (Fig. 10) a weighed portion of fuel is taken and mixed with a suitable proportion of a mixture of potassium chlorate and potassium nitrate (3 parts chlorate to 1 of nitrate), this supplying the oxygen necessary for the combustion of the fuel. After thoroughly mixing, the mass is transferred to the small copper cylinder A and a fuse inserted. (The fuse is made from cotton wick strands, soaked in potassium nitrate solution and dried.) The copper cylinder fits on a base B, on which three spring-clips are fixed. Over all a copper

cylinder C is secured by these clips, and a tube D with a tap attached forms the outlet. The whole apparatus is contained in a glass cylinder E.

In making a test a known quantity of water, usually 29,010 grains, is taken, and the temperature carefully noted by a thermometer graduated in tenths of a degree Fahrenheit. Thirty grains of the fuel mixed with ten to twelve times its weight of the oxidising mixture are placed in the smaller cylinder, which is fixed in position. The fuse is inserted, lighted, and the outer cylinder immediately placed in position and secured by the clips. The whole apparatus is immersed in the water before ignition takes place, the tap being of course closed. The products of combustion pass through the small holes at the bottom of the cylinder, and give up their heat to the water. When all evidence of action ceases, the tap is opened and water allowed to enter the copper vessel, taking up the heat from the inside portions of the apparatus. The whole is shaken up thoroughly, and the temperature of the water again carefully noted. The apparatus itself takes up a certain amount of heat, in proportion to its specific heat, and usually a correction of 10 per cent. is made for this.

Example :—

Weight of fuel taken - - - -	30 grains.
Weight of water taken - - - -	29,010 „
Temperature before ignition - - -	60·5° Fahr.
Temperature after ignition - - -	73·8° „
Rise in temperature - - - -	13·3° „

Then :—

30 grains fuel have increased temp. of 29,010 grains water 13·3° Fahr.
1 „ „ will increase „ $\frac{29010}{30}$ „ „ 13·3° „
1 lb. „ „ „ 967 lbs. „ 13·3° „

i.e., 1 lb. fuel in burning gives off 967 × 13·3 British Thermal Units
= 12861·1 B.T.U.

Allowing 10 per cent. for loss, we get a calorific power of
12,861 + 1,286 or 14,147 B.T.U.

The figure 29,010 is used as being 967 times the weight of fuel. By looking at above calculation, it will be seen that the calorific power is 967 × rise in temperature. As it requires 967 heat units to convert 1 lb. water at 212° Fahr. to steam, the number

of lbs. of water evaporated (or converted into steam) will be $\frac{967 \times \text{rise in temperature}}{967}$ or the rise in temperature + 10 per cent. will give at once the evaporative power of the fuel. Thus in above case the evaporative power is 13·3 + 1·3 lbs. of water at 212° Fahr. evaporated by 1 lb. of fuel, *i.e.*, 14·6.

In the William Thompson calorimeter, instead of using a solid oxidising agent, the gas oxygen is supplied either from a gas-holder or a cylinder. The combustion is started, as in the other case, by means of a short piece of fuse, or by a short platinum wire in an electrical circuit, which becomes hot enough to ignite the fuel when a current of electricity is passed through it. The coal or coke may be contained in a small platinum capsule, but in our experience this is not desirable, since the continued contact with hot carbonaceous matter renders the metal brittle in time; small porcelain crucibles answer equally well, in fact we have often used the bowl of an ordinary clay pipe from which the stem has been cut off, loosely plugging the bottom with a layer of asbestos. The measurements of temperature and calculation of results are carried out as before. It is desirable to note the time occupied by the combustion, and afterwards take the fall in temperature experienced by the water during the same length of time. A correction for radiation is thus obtained.

The Bomb Calorimeter.— In this type of calorimeter (Fig. 11) the combustion of the fuel goes on in a strong metal vessel, made of steel or some special alloy, which is in some cases lined with enamel, in others plated with gold inside, to resist the corrosive action of the gases produced, and often nickel-plated outside. Oxygen gas is supplied to this vessel, which is called the bomb or grenade, at a pressure of about twenty-five atmospheres. The fuel is placed in a small platinum crucible or capsule, over which is suspended a short piece of iron or steel wire. On the passage of an electric current through it, this wire becomes white hot and burns; the drops of molten magnetic oxide falling into the fuel ignite the latter, and the combustion continues until all the fuel is consumed. In some cases platinum wire is used to commence the operation. The bomb has a tightly-fitting lid, screwed on to a leaden washer. It is immersed in a

vessel containing water, and the whole is contained in a case lined with non-conducting material to prevent any loss of heat by radiation. The heat of combustion is transferred through the bomb to the water surrounding it, the increase in tem-

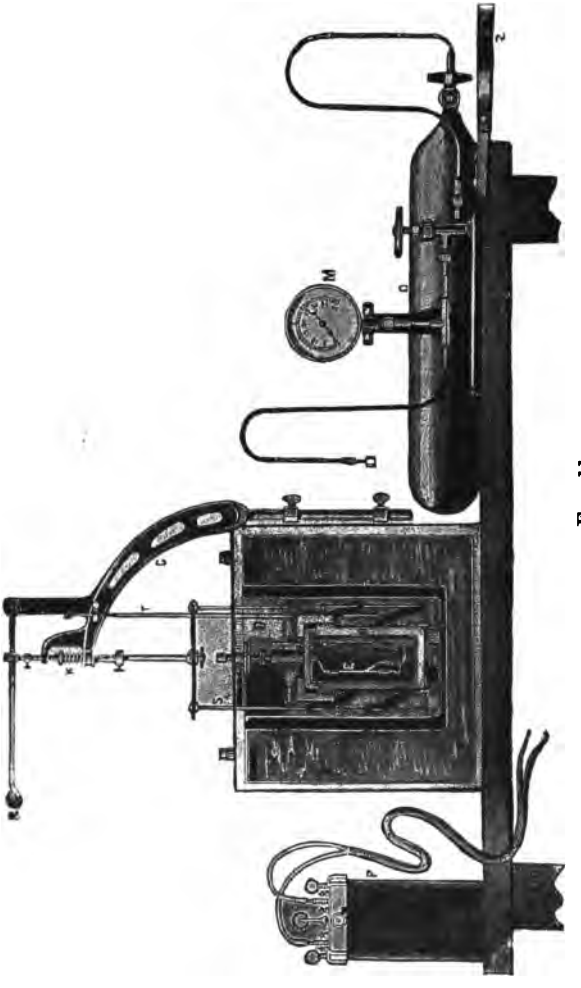


FIG. 11.
The Bomb Calorimeter.

perature noted, and the calculation worked out on similar lines to those previously given, corrections peculiar to the instrument being allowed for.

The illustration shows the Mahler bomb calorimeter, with

the necessary appliances. The cost of the bomb type of calorimeter is considerably greater than that of Lewis Thompson or William Thompson class. The former may be anything from £10 to £50; the latter from £2 to £5.

By means of these various calorimeters, it will be seen that the heat produced by the absolutely complete combustion of the fuel is measured. Practically, the most satisfactory method of testing the quality of various coals is to make an evaporative test under actual working conditions. The burning qualities of the coal can then be noted also, as well as the kind and amount of clinker and ash which are produced. To make a test on these lines requires careful and special arrangements, and occupies much time. Hence the use of a calorimeter of some type is necessary, and there is no question that such instruments afford reliable comparative data.

An average calorific value for coal is about 7,000 calories, that of coke somewhat less. It follows from this that 1 lb. of good coal should be able to generate 12 to 14 lbs. of steam. In actual practice, if 6 to 8 lbs. of steam are obtained per 1 lb. fuel, it is as much as can be expected. There are several factors which account for the difference between a calorimetric test and the practical results. Loss of heat by radiation; the impossibility of regulating the admission of air so as to supply exactly the amount required for the perfect combustion of the fuel. If too little, imperfect combustion occurs with production of smoke and carbonic oxide and consequent loss of heat; if too much air then the excess uses up some of the heat in raising its own temperature to that of the other gases, which heat would otherwise be used for steam generation. Again, injudicious or careless firing leads to smoke and carbonic oxide production and loss of heat, and the capacity of gases for heat increases with their temperature.

It is possible to calculate the heating value of a fuel from its chemical composition, knowing the carbon, hydrogen, oxygen, nitrogen, sulphur, ash, and moisture. The substances which produce heat when a fuel burns are chiefly carbon and hydrogen, and sulphur to a very slight extent. Any oxygen which may be present causes a loss of heat, because it uses up its equivalent amount of hydrogen, leaving only the excess as a heat producer. Moisture in the coal, together with that produced by the com-

bustion of any oxygen present, takes up heat in its conversion into steam, and of course the mineral matter or ash is a non-producer of heat. Calculated heating values do not as a rule agree very closely with practical tests, or even with calorimetric results.

CHAPTER VI.

Coke Ovens (1).

Coking.—As stated in previous chapters, some coals are not suited for use in blast furnaces or foundry cupolas owing to the caking properties. The caking is due to the constituents of the volatile matter of the coal, and the manner in which these constituents are grouped. If, however, a coal possesses caking properties, it may be rendered suitable for the above processes by subjecting it to heat under certain conditions. The heat drives off the volatile matter and leaves the fixed carbon, which, along with the ash or mineral matter, forms coke. In olden times the coal was heated or coked in mounds or heaps in the open air. This method was certainly crude, and gave place to the beehive or enclosed type of oven, from which the air was partially excluded. The beehive oven is now being superseded very largely by the retort or closed oven type, in which the bye-products are extracted. The open mound process is practically done away with, owing to its wastefulness. The beehive oven still possesses the advantage over the retort type in the lower prime cost and simplicity of working, requiring, as it does, little skilled labour, whilst producing excellent coke. These advantages, however, are more than compensated for, in bye-product ovens, in the saving from bye-products, as also from the spare power now obtained in modern plants through a more economical use of the gas from the ovens. Before dealing with bye-product ovens of recent construction, a brief description of the older types of ovens will not be out of place, as showing the lines of progress in coke oven design. The earliest type of coke oven is the beehive oven, the general shape of the oven being indicated by the name. The interior dimensions of this oven are usually about 12 feet in diameter and 7 feet high. The inner lining is of

refractory brickwork, and the space between this lining and the outside walls is filled with sand, slag, or brickbats, to retain the heat. As shown in Fig. 12, they are usually built in double rows, each oven being connected to a common flue, the connection being controlled by a damper. The waste heat from this flue passes through boilers to the chimney. The operation of coking is carried on as follows:—The doorway is partially built up, and the charge of slack fed into the oven from hoppers. Sometimes a small fire at the door is necessary to start the action, but when in full working order, the heat retained in the oven brickwork is usually sufficient. The charge is levelled and the door built up. For a few hours the gas comes off slowly, and is not of a quality to ignite, but after a short time the gas comes off more freely, and at this stage a little air is admitted above the charge

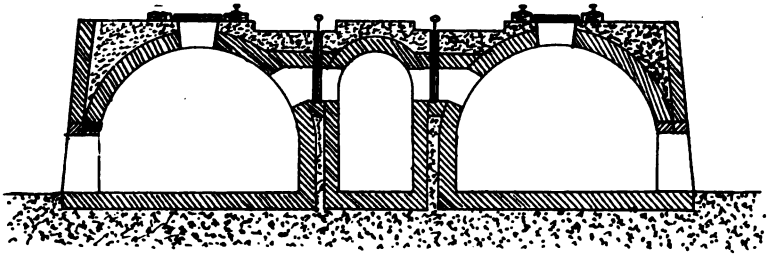


FIG. 12.—Beehive Coke Oven.

to burn the gas, the heat from this combustion being reflected by the roof on to the charge below. The heat of the oven gradually increases, and the whole of the volatile matter is finally expelled. This requires about three days, and during the last portion of the coking the door is thoroughly luted to exclude air entirely, to avoid undue loss of coke. The yield of coke in these ovens, using slack containing 30 per cent. volatile matter, ranges from about 56 to 60 per cent., showing a loss of 10 to 14 per cent. through admission of air to the coking chamber. When all trace of gas is absent, the mass of incandescent coke is quenched inside the oven, involving a further waste in heat, and the charge is then raked out. To avoid this cooling of the oven itself by quenching, modifications of the oven have been built in which the charge may be drawn out by mechanical means and quenched outside; but a more im-

portant saving was effected by the introduction of the retort type of oven by Coppée in 1861, the improvement consisting in complete exclusion of air from the coking chamber. The simplest type of this oven is shown in Fig. 13. The ovens shown consist of long rectangular chambers about 30 feet long and 3 feet 6 inches high. They are usually built with a slight taper in the width, being about 15 to 17 inches wide at one end

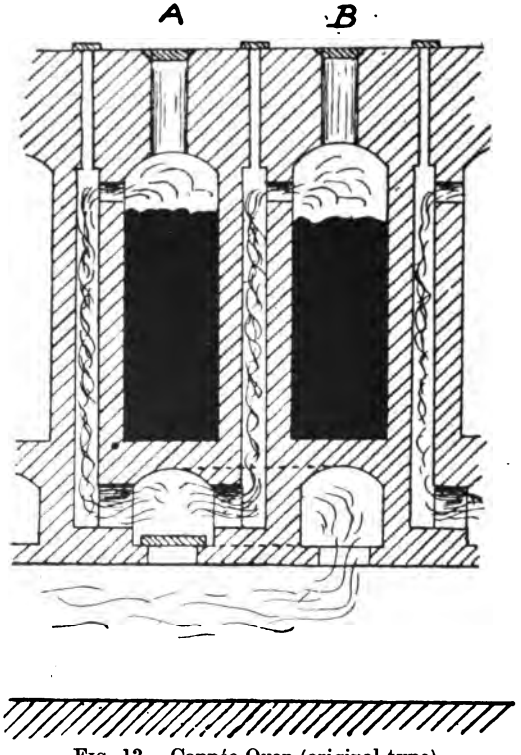


FIG. 13.—Coppée Oven (original type).

and 18 to 20 inches at the other. This is to lessen the friction of the charge on the oven walls whilst discharging. The ovens are charged from the top, and the gases from the charge pass, as shown, into a series of vertical flues, in which is also admitted the air necessary for combustion. This air is in some cases heated to a temperature of 700° Fahr. by being conducted through passages in the hot brickwork underneath the ovens.

The heated products of combustion pass downwards into a sole flue, and then passing under the whole length of one oven return by the sole flue of the adjoining oven, finally passing

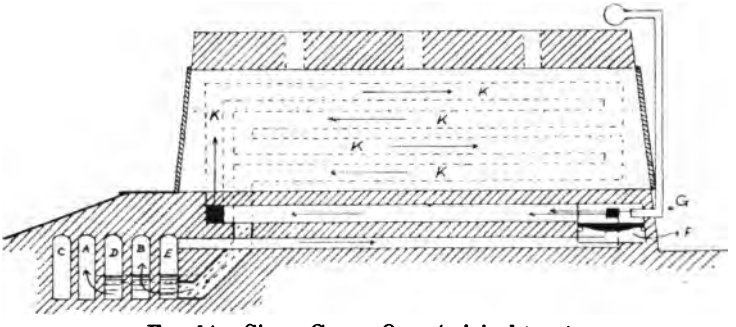


FIG. 14.—Simon Carves Oven (original type).

into the chimney flue, passing under boilers before reaching the atmosphere. When the volatile matter has been completely expelled, the charge is pushed out by a ram and quenched outside the oven. The advantages gained in this oven are:—Increased yield, through exclusion of air; shorter coking period through the narrower chambers in use and the more efficient means of utilising the gases; saving in the heat of the oven through external quenching; saving of time and labour through the use of mechanical appliances, &c. This oven has, of course, been adapted for the recovery of bye-products, but the original described is sufficient to show that from it a great many of our modern coke ovens have arisen.

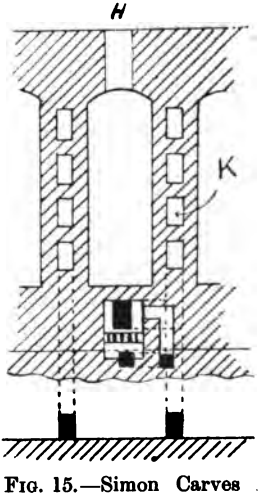


FIG. 15.—Simon Carves Oven (Vertical section).

The Simon Carves oven (Figs. 14, 15) was the first retort oven to be erected in England for the recovery of bye-products, a battery of these ovens being erected in this country about 1869, and it is from this Simon Carves oven that all the other types of horizontal flued ovens have been developed.

The gases are drawn off at H and, by means of an exhauster, passed through the bye-product plant and returned to the distributing main M. From this main, through a service pipe for each oven, the gas is fed above a firegrate underneath the floor

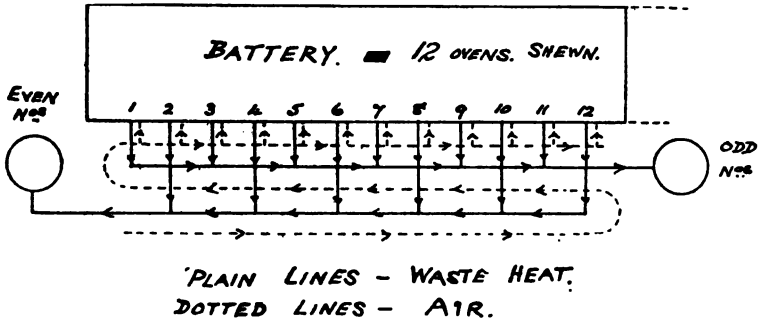


FIG. 16.

of the oven. Here a supply of air, preheated by a simple regenerative system to about 800° Fahr. (or 425° Cent.), is admitted. Combustion takes place, and the heated products pass along the sole flue, thence to the top side flue, thence in a

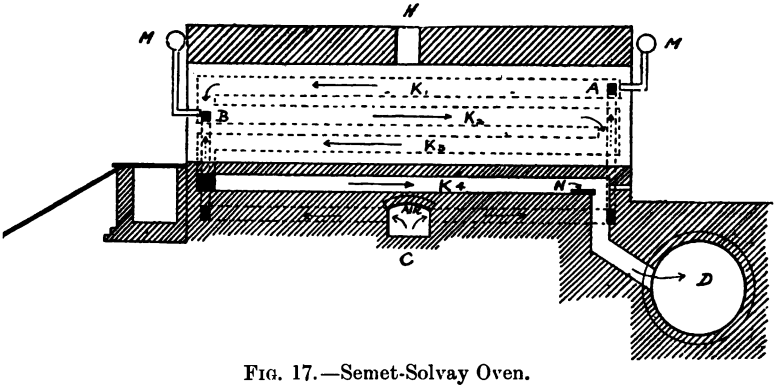


FIG. 17.—Semet-Solvay Oven.

zig-zag direction to the recuperative system of flues. This system is shown diagrammatically in Fig. 16. From the diagram it will be seen that the waste gases from odd-numbered ovens pass to a chimney at one end of the battery, whilst a chimney at the opposite end of the battery extracts the waste

heat from even-numbered ovens. The air flues are sandwiched between these two flues.

The Semet-Solvay coke oven in principle is somewhat similar to the Simon Carves. The diagram (Fig. 17) shows the usual arrangement of flues. Some of the existing ovens have three flues at each side, as in Figs. 17, 18, but the tendency now is to

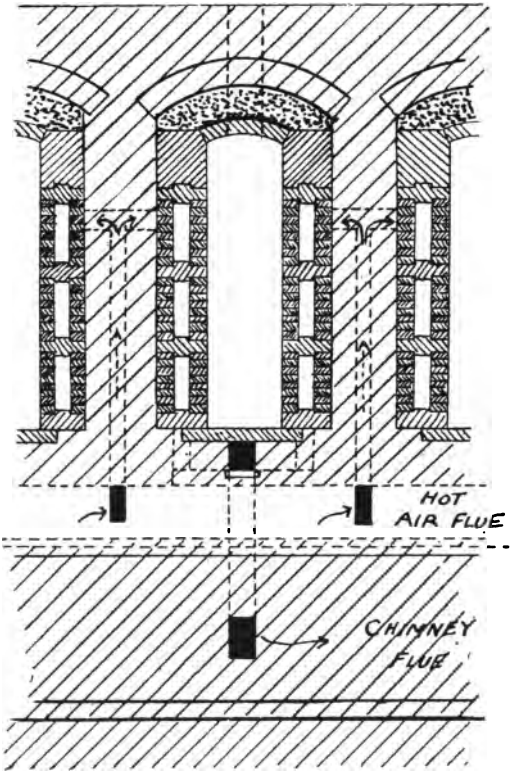


FIG. 18.—Semet-Solvay Oven (Vertical section).

adopt four or five side flues, as in Fig. 19, the ovens being of correspondingly greater height. This is an undoubted advantage, as modern compressing machinery will charge a larger oven just as readily as a smaller, whilst the larger oven takes up only the same area as the smaller. The standard Semet-Solvay oven is of strong, substantial design. The oven side flues are entirely independent of the superstructure, the weight

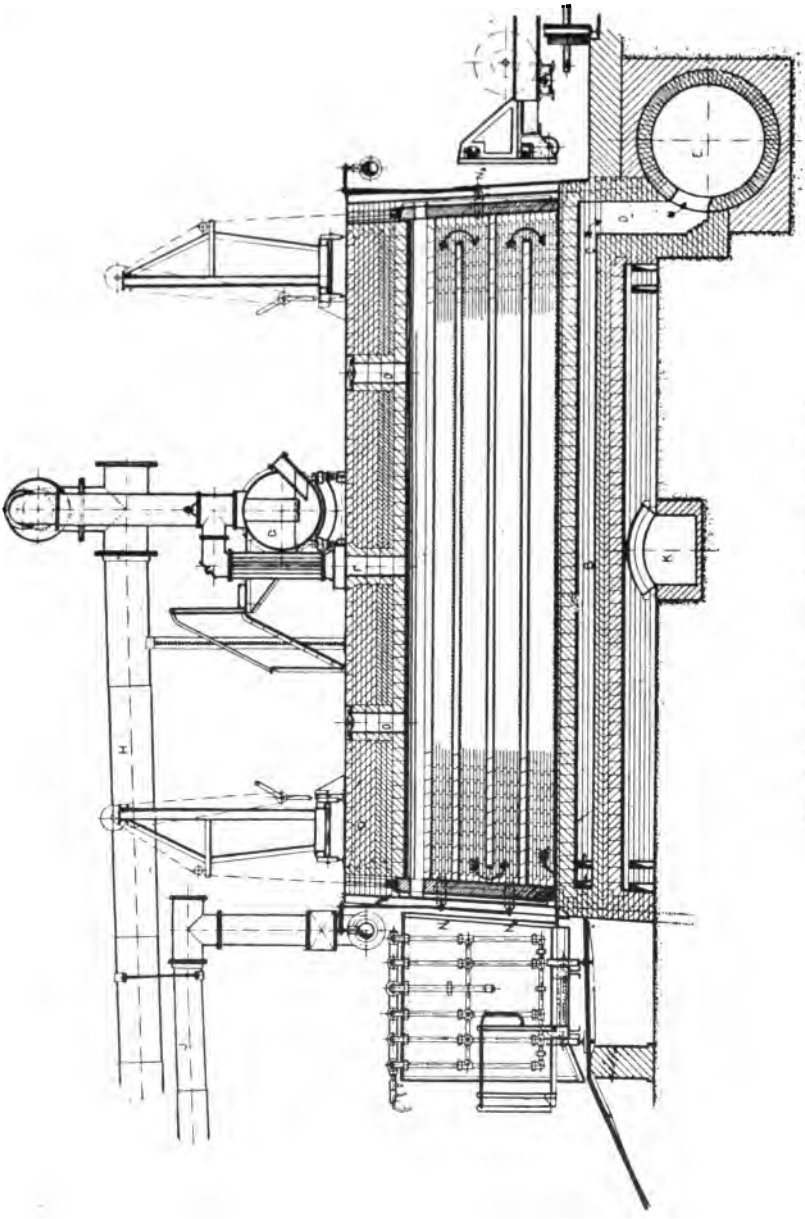


FIG. 13.—SEMET-SOLVAY OVENS (four flues).

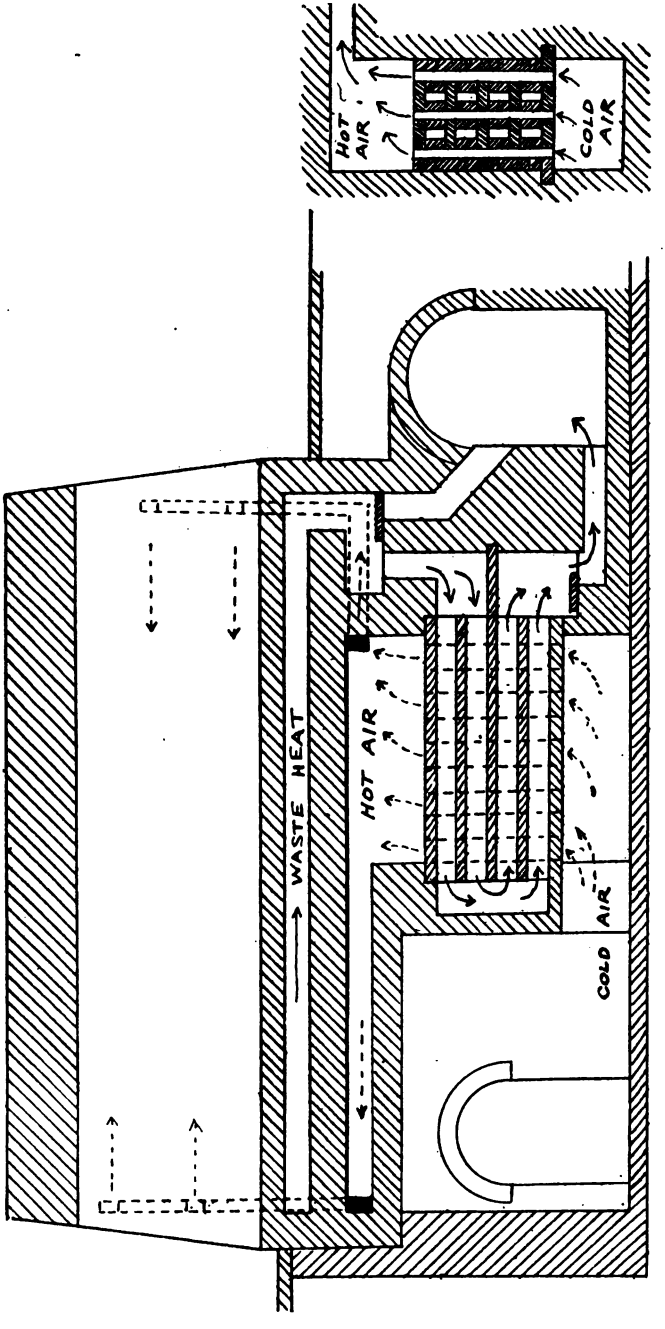


FIG. 20.—CONTINUOUS REGENERATOR FOR SEMET-SOLVAY OVENS.

FIG. 21.—SEMET-SOLVAY COKE OVENS, LAMBTON COLLIERIES, LTD.

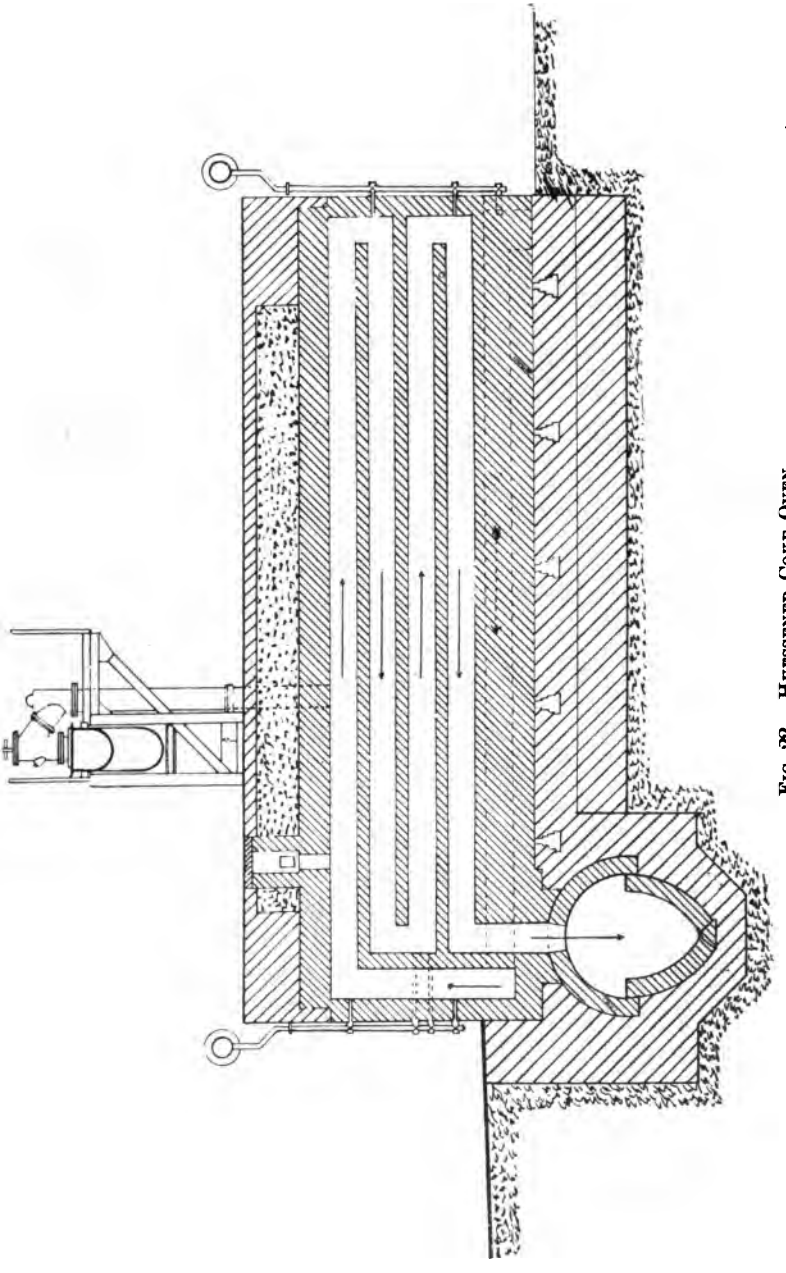


FIG. 22.—HUESSENER COKE OVEN.

of which is carried on separate pillars. The brickwork of these pillars is built of less refractory but tougher material than that of the flues themselves. As well as materially strengthening the oven, this pillar acts as a reservoir of heat, a great advantage during temporary stoppages of the plant. The heat stored in the pillar also assists in counteracting the cooling of the flues through the introduction of a charge of wet slack.

It will be noted from Fig. 18 that in the Semet-Solvay oven each oven is entirely independent of its neighbour, having two series of side flues instead of a series of flues common to two adjoining ovens (as is the case in the majority of retort ovens). The heat of the oven is thus more readily controlled, and any single oven may be repaired without interfering with the working of the two adjoining ovens. To ensure gas-tightness, a very important item indeed, the flues are usually built of small rectangular blocks about $8 \times 4 \times 2$ inches. Larger blocks, owing to the expansion and contraction during charging, &c., tend to open at the joints, allowing a portion of the gas to pass into the side flues, the bye-products in this leakage being, of course, lost, besides decreasing the amount of "spare gas" from the battery. These small bricks can readily be obtained with very uniform surfaces, and the jointing need not, with ordinary care, be more than $\frac{1}{8}$ inch in thickness. The gases from the oven, after being passed through the bye-product plant, return to the distributing main M, from which it is fed into the flues as shown. In the ordinary type of Semet-Solvay oven the air for combustion is preheated, by being drawn through passages underneath the oven, to about 300° Cent., but a later type of continuous regenerator has been introduced. This type of regenerator is shown in Fig. 20. It possesses the great advantage of being continuous in action, thus avoiding the sudden changes

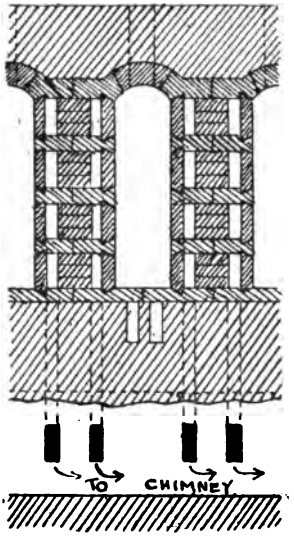


FIG. 23.—Huessener Oven (Vertical section).

of temperature during the periodical reversing which takes place in the ordinary type of regenerator. At the same time the regenerator may be shut off for repairs quite easily without affecting the oven itself, using cold air in the meantime.

Referring to heating arrangements of the oven, the gas is fed into the top side flues K_1, the hot air coming into the side of this flue from a passage in the partition wall. This air is controlled by a slide damper at A. Passing along the flue, the gas is reinforced at B, where air is also again introduced. The supply of gas to each of these places is carefully regulated by diaphragms placed in the service pipe, perforated to a definite size determined by the usual conditions of working (*i.e.*, chimney draught and pressure of gas). In three-flued ovens it is not necessary to introduce more gas, as the products of combustion from K_1 and K_2 are quite sufficient to maintain a good heat in flues K_3 and K_4, as shown by the following actual temperatures:—

Top side flue, K_1 - - - - -	1,190° Cent.
Middle side flue, K_2 - - - -	1,200° „
Bottom side flue, K_3 - - - -	1,170° „
Sole flue, K_4 - - - - -	1,100° „

In four-flued ovens there are usually two gas supplies at each end of the oven. The condition of these flues can be noted through inspection holes at each end, from which the whole length of the flue may be seen, and very uniform heats may be obtained. The Semet-Solvay ovens are usually charged by machinery which compresses the charge before placing it in the oven. There are various advantages derived from the use of compressed charges, and these will be discussed later. The amount of spare gas is, in the ordinary type, about 30 per cent., and with continuous regenerators about 50 per cent.

The Huessener coke oven (Figs. 22, 23) is also of the horizontal flued type. The arrangement of flues is somewhat similar to the original Simon Carves oven, but, like the Semet-Solvay, each oven has its own side flues, the ovens being thus entirely independent of each other. The flues are constructed of refractory tiles and bricks well dovetailed together. The air for combustion is not preheated. The gas and air supplies are well under control, and good heats are obtained in the flues, as

FIG. 24.—HUESSENER COKE OVENS, MARLEY HILL COLLIERY, DURHAM.

shown by the temperatures given in a paper read before the Iron and Steel Institute in 1904.*

Bottom side flue - -	1,900° Fahr.,	1,037° Cent.
Top side flue - - -	2,200° „	1,205° „

The waste heat passes under boilers, evaporating 24 cwt. of water per ton of coal, and the spare gas amounts to 30 per cent. of the gas generated. This oven is also designed on the regenerative principle if desired. The system of bye-product recovery now being adopted in connection with the above plant is described later.

* C. Lowthian Bell, Iron and Steel Institute, 1904.

CHAPTER VII.

Coke Ovens (2).

The Otto-Hoffmann coke oven (Fig. 25) has been adopted to a very large extent in America. Like the old Coppée oven, it is heated by vertical flues, but differing in this respect, that no direct communication exists between the interior of the oven and the side flues when working under normal conditions, the

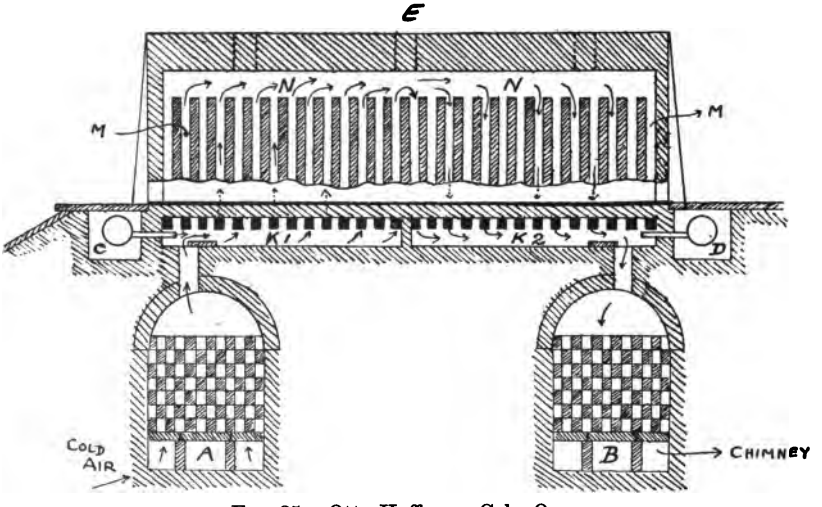


Fig. 25.—Otto-Hoffmann Coke Oven.

gases being conducted through the bye-product plant before admission to the side flues. The air necessary for combustion is preheated very considerably in regenerators. As this system of regenerators is common to several types of coke ovens, a brief description will be given. Underneath the sole of the oven are two combustion chambers, K_1 and K_2, fed by gas

supplies at C and D respectively. The gas is only supplied to one combustion chamber at a time, the illustration showing chamber K_1 receiving gas from main C, whilst the gas cock at D is closed. The air for the correct combustion of gas in K_1 is supplied through the chequered brickwork at A, usually by means of blowers. The gas burns in K_1, and the heated products of combustion pass upwards through one-half of the vertical flues M into the upper combustion chamber N, thence downwards through the other half of these flues, thence through the combustion chamber K_2 to the regenerators at B, and thence to the chimney. These heated waste gases give up their heat to the brickwork at B, raising the temperature of the bricks to a full red heat. Then, if the direction of the gas and air be reversed (about every half-hour), the air being blown through regenerators B is raised to a temperature of 900° to 1,000° Cent. Being so heated, a considerably less quantity of gas is required to keep up the temperature of the flues M, and consequently more spare gas is obtained for use in gas engines, or for lighting purposes. Of course, where regenerators are used all the heat, or nearly all, is extracted from the waste gases, leaving no heat available for steam raising by means of boilers. The choice, then, for oven builders lies between waste heat or steam power, and live gas or gas engines. On the one hand is the cost of boilers and the loss in efficiency as opposed to gas engines. On the other hand is the cost of regenerators and a gas-holder, which, if not absolutely necessary, is advisable, having regard to the irregular supply of gas from a battery of coke ovens. In addition, the cost of extra purification of the gas should be included. One drawback to the use of waste heat under boilers is rather important. The waste heat must be used immediately and actually on the spot, as the waste heat flue cannot be carried any great distance without serious loss of heat. Also, should surplus power not be required for the time being, the waste heat must be bye-passed to the chimney. Considering the rapid strides made in gas engine construction, and the possibility of the more general use of coke-oven gas for lighting purposes, the ovens on the regenerative principle are now coming more into vogue, but at the same time most oven builders retain a waste heat type for those who prefer a well-tried servant in the form of steam, to a newer servant in the form of gas.

The Otto-Hilgenstock oven as now built, is a distinct improvement on the Otto-Hoffmann oven. It is built in two types, "waste heat" or "live gas," shown in Figs. 26, 26*a*, 27, and 28 respectively. It will be seen that in both cases the vertical flue has been retained, but the distribution of the heat has been vastly improved. The gases from the oven chamber are conducted through the bye-product plant, the most recent type of which is shown in Fig. 94. After being deprived of the bye-products, the gas is led by branches from a distributing main M into a series of nozzles or Bunsen burners N. In the case of the "waste heat" oven the action of the Bunsen burners draws in sufficient air for the combustion, this air being heated in its passage to the combustion flues. In the case of the "regenerative" type, the air is preheated by passing through the regenerators K, afterwards being conducted underneath the sole of the oven to the combustion chambers F through the ports R. In both cases the gas, instead of passing into one combustion chamber, is subdivided, being fed into the vertical flues in as many as fifteen or sixteen places. Thus, each burner is required to heat only a small portion of the oven walls, and, as the air and gas supply are perfectly under control, sooting of the flues through deficiency of air, or fusion of the flues through excess, is easily avoided, and the life of the oven walls is prolonged considerably.

The gas ignites at the level of the coking chamber, and rises vertically through the heating flues, following its natural tendency. The chimney draught is thus decreased, and the loss of gas, through leakages from the oven chamber to the side flues, is reduced to a minimum. In the "waste heat" type the amount of spare gas is 20 to 40 per cent., but by using regenerators the amount of spare gas is increased to 50 per cent. The ovens may be charged by tubs from the top, as shown in the drawings, Figs. 26, 27, but the commoner plan is to use compressing machinery, as described in Chapter IX. By using the latest type of ammonia recovery plant, as shown in Fig. 94, the amount of steam required for the actual working of the plant is considerably reduced, and the steam available for outside purposes correspondingly increased.

The latest type of Simon Carves oven is a good example of the modern tendency in vertical flued ovens; entire control

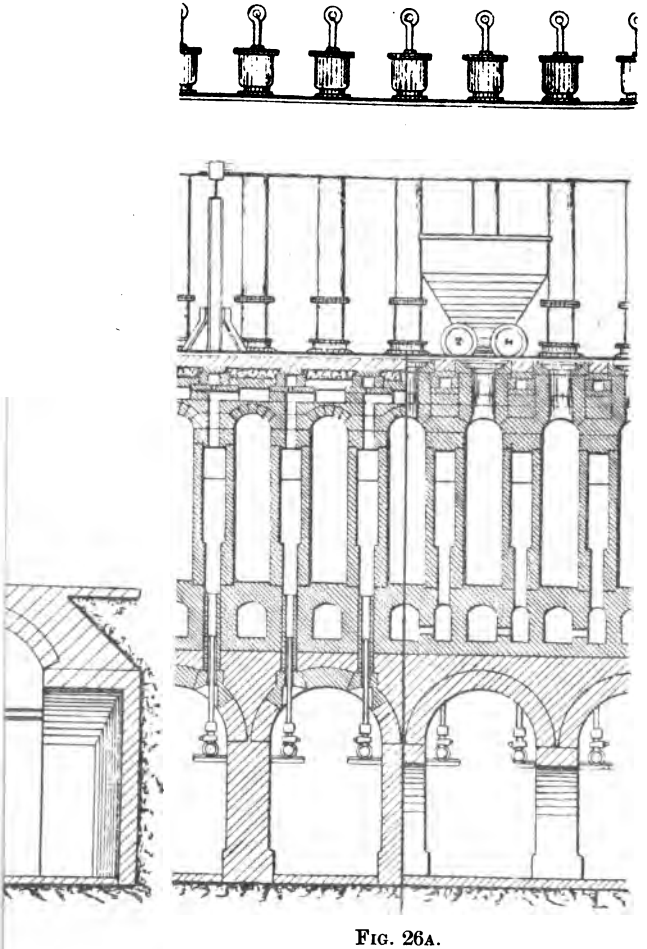


FIG. 26A.

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FIG. 29.—OTTO-HILGENSTOCK COKE OVENS, CRIGGLESTONE COLLIERY (Charging side).

FIG. 30.—OTTO-HILGENSTOCK COKE OVENS, CRIGGLESTONE COLLIERY (Coke Bench side).

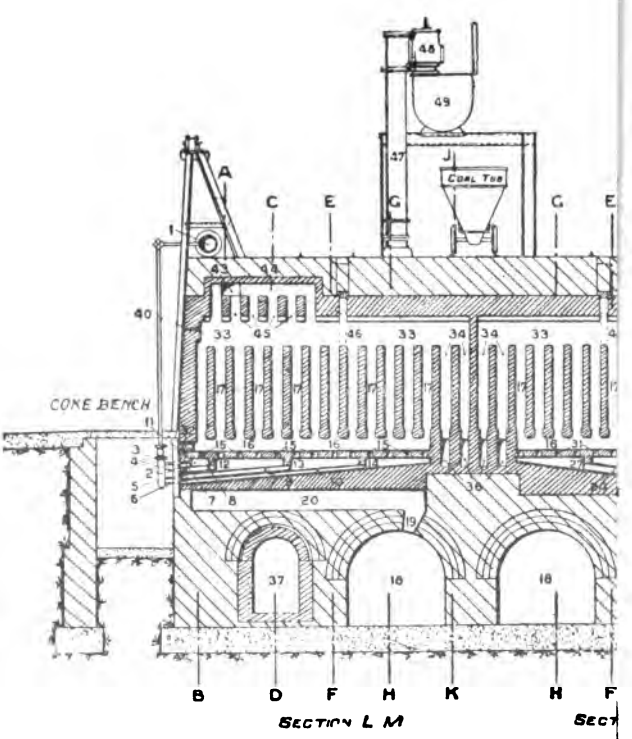

FIG. 33.—SIMON CARVES COKE OVEN. (Wast

over the supply of air and gas being attained, and this regulation can be effected in the most easy manner, as all cocks and dampers are operated from the outside (see Fig. 35).

As seen in the sketch (Fig. 31), the flues are in two sections, fed alternately by the gas jets Q_1 and Q_2, and the supply of gas is subdivided into ten sections, giving a uniform heat over the full length of the oven pillar. The admission of gas to each of these ten sections is regulated by ten specially designed cocks, and the supply of air is likewise divided up into ten sections corresponding with the gas supply, the admission of air to each section being regulated by dampers.

These gas-regulating cocks, and also the dampers for air

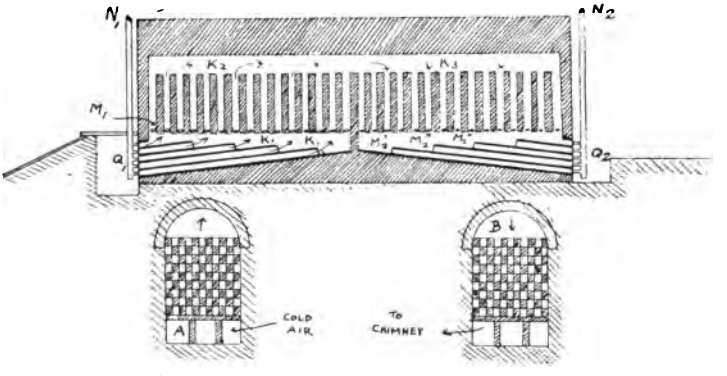

FIG. 31.—Simon Carves Oven (Section of flues, &c.).

supply are all accessible from the ram track and coke bench and it is entirely unnecessary to enter the hot-air galleries under the ovens.

The inlets from these chambers into the vertical flues are shown at M_1 and M_2 on the drawing. Thus, under the conditions shown, gas is being fed into the flues by the firebrick tubes K_1. The air enters at M_1 from the regenerator A. The products of combustion pass from K_1, K_2, K_3 through openings M_2 into regenerator B, and thence to the chimney, the gas cock at N_2 being closed during this period. This oven is also adapted as a "waste heat" oven, as shown in Figs. 33, 33*a*. The regenerative type is shown in Figs. 32, 32*a*, 32*b*.

FIG. 34.—SIMON CARVES OVENS, WHARNCLIFFE WOOD MOOR COLLIERY, BARNSLEY.

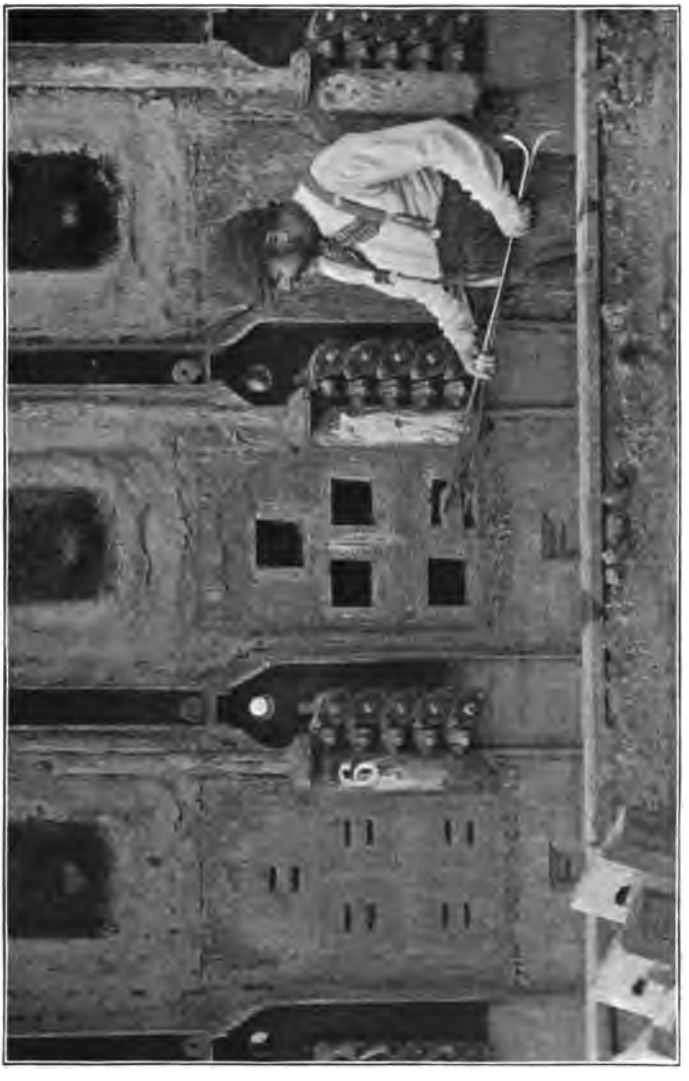

FIG. 35.—REGULATING AIR-DAMPERS IN SIMON CARVES REGENERATIVE OVEN.

CHAPTER VIII.

Coke Ovens (3).

The Koppers coke oven is of the vertical flued type, possessing many striking features in the heating arrangements of the flues. The oven is built in two types—waste heat and regenerative. The waste heat oven (Figs. 36 to 41) is of very simple construction. The gas, after being deprived of its bye-products, is fed from the distributing main C into the gas-distributing channel E, formed of firebrick pipes. The gas passes from this channel through orifices, each fitted with a gas nozzle, into the vertical flues. These flues, numbering from thirty to thirty-five, have each a separate nozzle, the details of which are shown in Fig. 38. The nozzles have oval-shaped orifices, and by means of a rod with a T end (Fig. 39) may be very easily changed. These nozzles have orifices of varying size, so that the amount of gas passing into each individual flue can be perfectly adjusted. The air necessary for combustion is drawn in by the chimney draught through the air-distributing channel G (Fig. 38) from the air conduit F. From this channel it issues by the ports H into the combustion flue K, flowing round the nozzle U, and keeping it comparatively cool. The amount of air is controlled by the unique damper arrangement shown in Fig. 37. As both dampers and gas nozzles may be adjusted from the top of the oven the combustion of gas in the flues can be easily controlled so as to maintain an exceedingly high state of efficiency, and the greater number of gas jets ensures a uniform temperature of the oven walls from end to end. The products of combustion pass into the waste heat flue through a passage regulated by the damper R (Fig. 36). The waste heat passes along the flue P and through the boiler tubes by an arrangement as shown at Fig. 42.

The regenerative type of Koppers coke oven is shown in Figs. 44 to 47. This is an improvement on the original

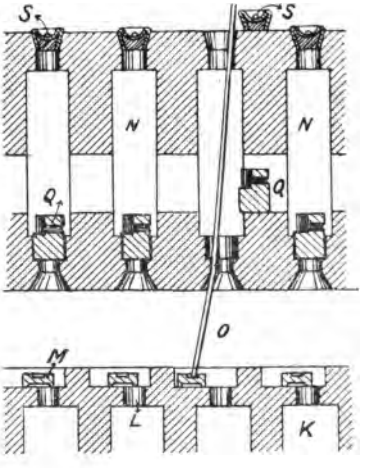

FIG. 37.

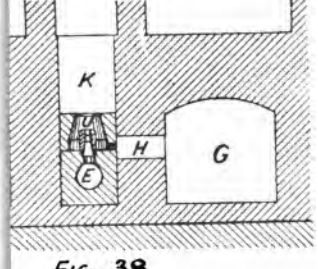

FIG 38.

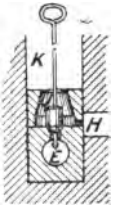

FIG. 39

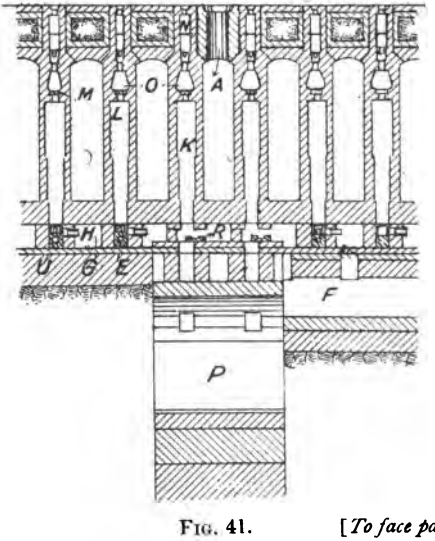

FIG. 41. [*To face page* 58.

FIG. 42.—TWELVE LANCASHIRE BOILERS FIRED BY WASTE GASES FROM 65 KOPPERS NON-REGENERATOR OVENS, AT MONT CENIS COLLIERY, WESTPHALIA.

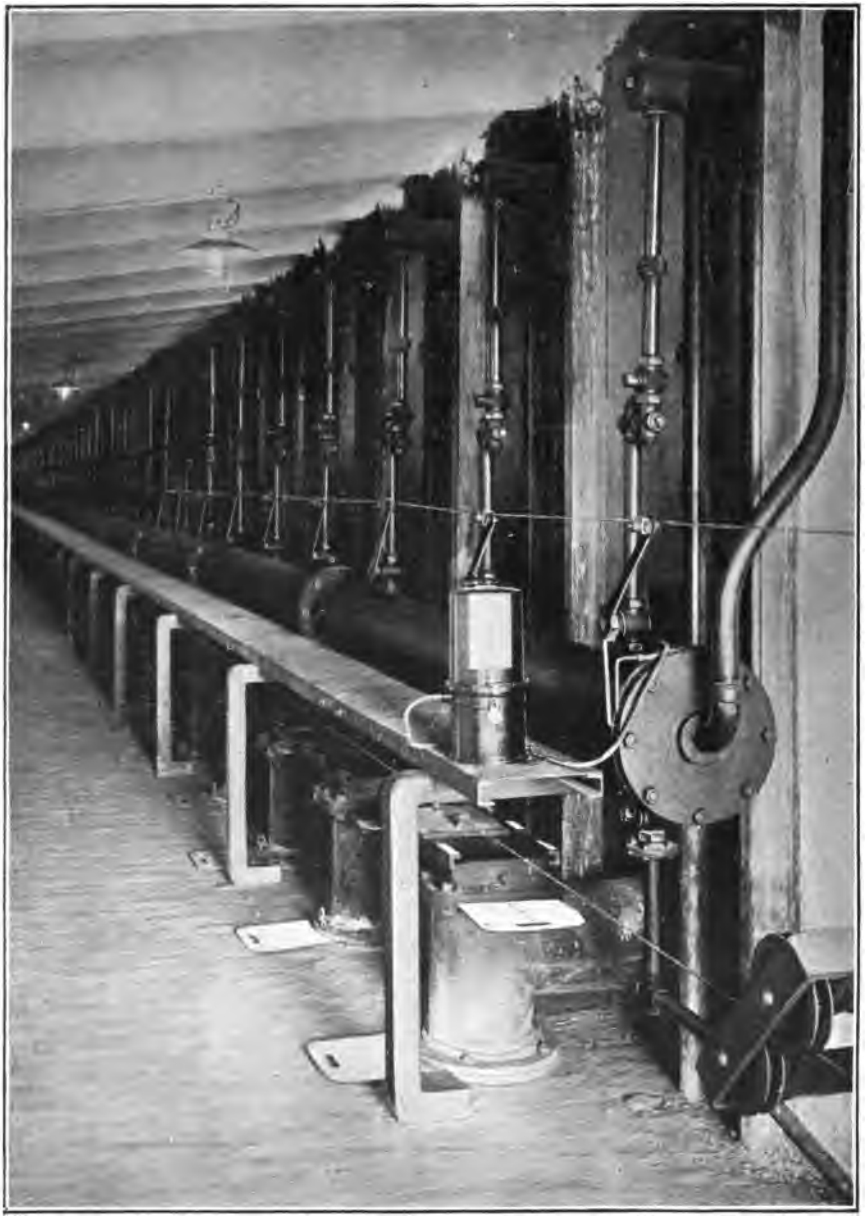

FIG. 43.—VIEW IN PASSAGE WAY SHOWING GAS AND AIR REGULATION FITTINGS.

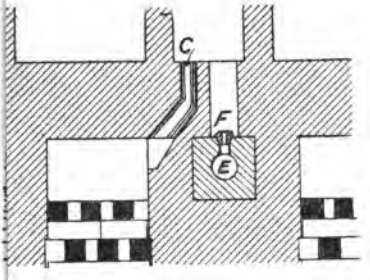

FIG. 45

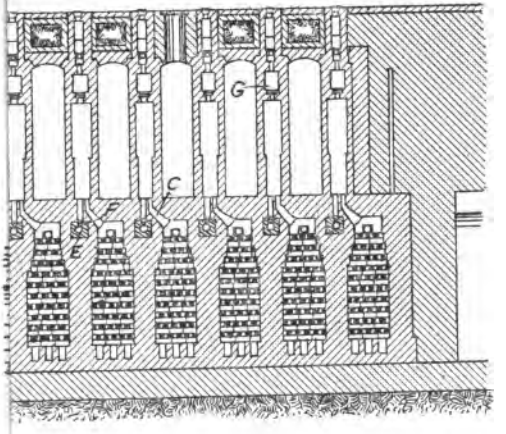

FIG. 47.

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FIG. 48.—KOPPERS COKE OVENS, BARNSLEY MAIN COLLIERY.

Koppers regenerative oven, in that each oven has an entirely separate regenerator, which allows of repairs to individual ovens without affecting the remainder of the battery. The reversing

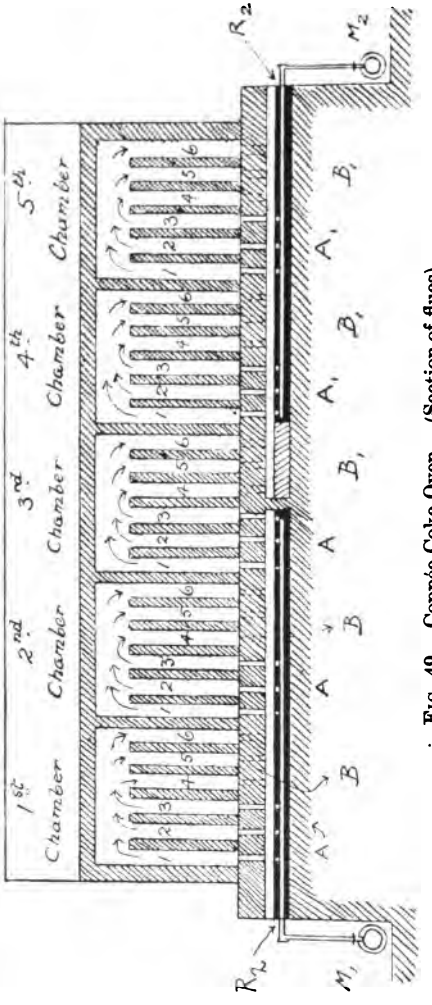

FIG. 49.—Coppée Coke Oven. (Section of flues).

of the gas and air is done simultaneously along the whole of the battery by a link arrangement shown in Fig. 43. It is found that the waste heat is more than sufficient to maintain the heat in the regenerators, and consequently arrangements are provided

for taking off a portion of the products of combustion through the flues T and V (Fig. 44). The arrangements for regulating the combustion in the individual flues are the same as described in the waste heat type. The amount of spare gas from ovens of the waste heat type varies from 15 to 20 per cent., whilst with regenerators the amount is from 50 to 60 per cent. of the total gas evolved.

The Coppée oven, as now designed, is the result of continued

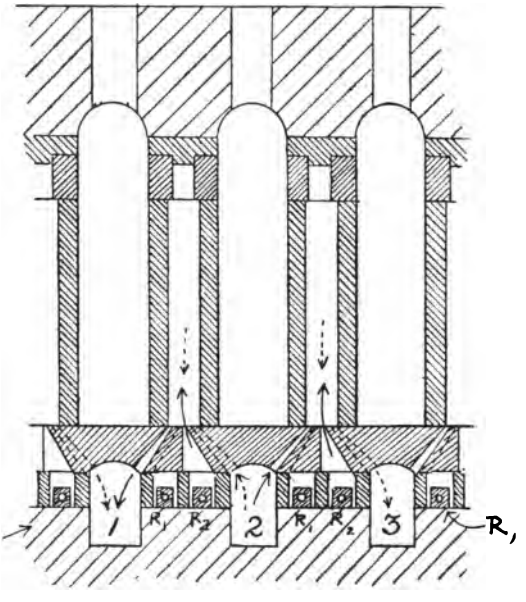

Fig. 50.

Coppée Oven (Section of flues).

improvements on the original Coppée oven described on page 38. The oven is now constructed for bye-product recovery and is designed on the "waste heat" or "regenerative" principle. The regenerative type is shown diagrammatically in Figs. 49, 50. The regenerators are situated underneath the ovens. One regenerator is connected to the sole flue of the odd-numbered ovens, whilst the other is connected to the even-numbered ovens. The series of vertical side flues is divided

into five sections, each consisting of six flues (Fig. 49). The gas is fed into the flues by means of the tubes R_1 and R_2, two at the front of the ovens and two at the back. The tubes R_2 feed the vertical flues marked 1, 2, and 3 in each chamber, whilst the tubes R_1 feed the flues marked 4, 5, 6. Thus for a period of about thirty minutes the gas cocks that feed R_2 are opened and the flues marked 1, 2, 3 are receiving the gas in consequence. The air, heated to a temperature of about 1,000° Cent., comes along the sole flue 2 (Fig. 50) and meets the gas at the foot of the vertical flues. The heated waste products pass down 4, 5, and 6 to the sole flues 1 and 3, &c. (Fig. 50), thence through the corresponding regenerator to the chimney. After this period the direction is reversed, R_1 then feeding flues 4, 5, and 6, and the sole flue 2 taking away the products of combustion from flues 1, 2, and 3, &c. By this arrangement the reversing of the air current affects only three flues at a time, or one-tenth of the oven wall. Thus the flues for the gases descending for the time being are nearly as hot as the ascension flues, and a uniform temperature is maintained throughout the length of the oven wall. The air supply is driven through the regenerators by means of a fan driven by electric motor, and is regulated by this means.

The Collin oven (Fig. 54, waste heat type, and Fig. 55, regenerative type) is of the vertical flued type, with or without regenerators. The principal feature of this type of oven will be seen from the section *l-m*, which shows a series of flues with bond stones between. These bond stones are hollow, and in the case of the regenerative oven the flues thus formed serve two purposes :—

1. To take off the waste gases after combustion in the main vertical flues.
2. To conduct hot air from the regenerators to the gas from the upper distributing channels during another period of the coking process.

The sectional area of these inner flues has been carefully predetermined to give the correct proportion of gas and air. The flues are worked in two stages as stated above. During the first stage the gas passes from the lower series of conduits into the base of the main flues, where it meets the hot air from

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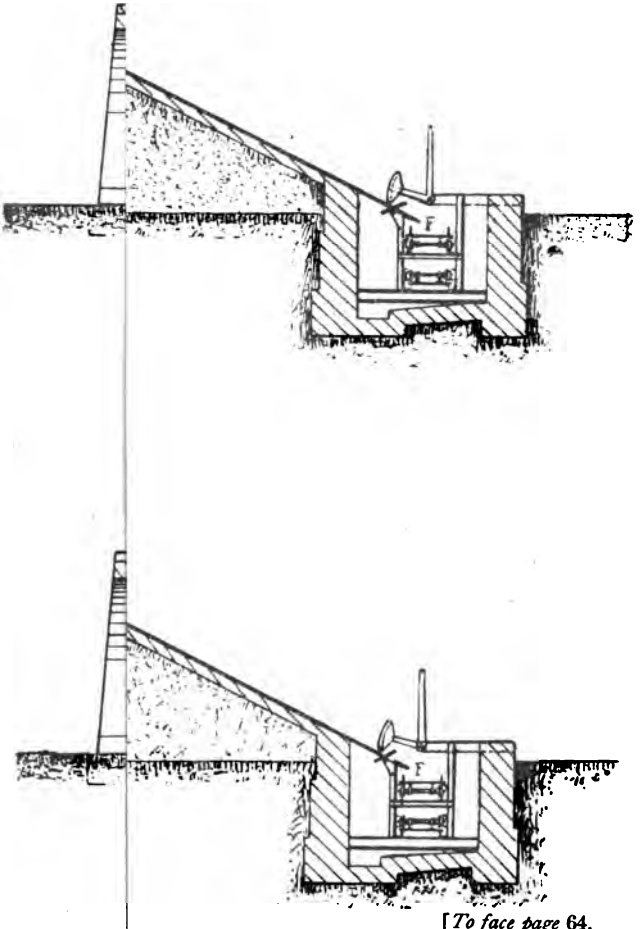

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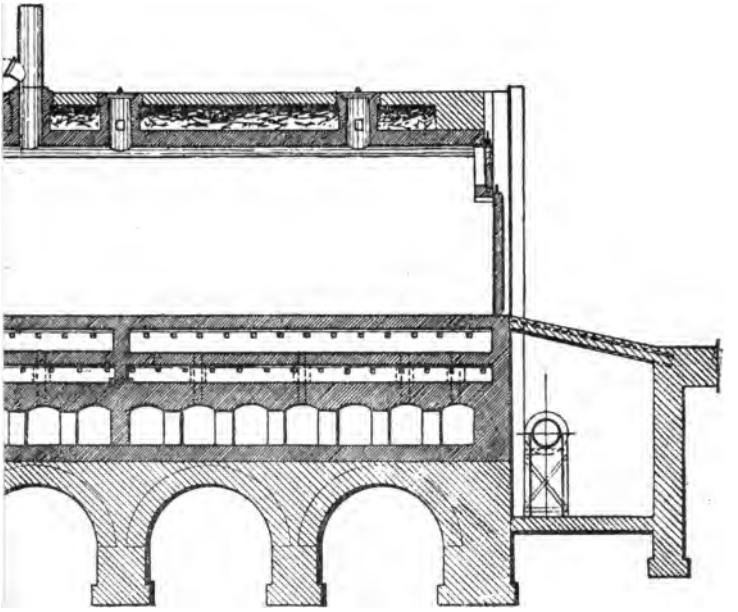
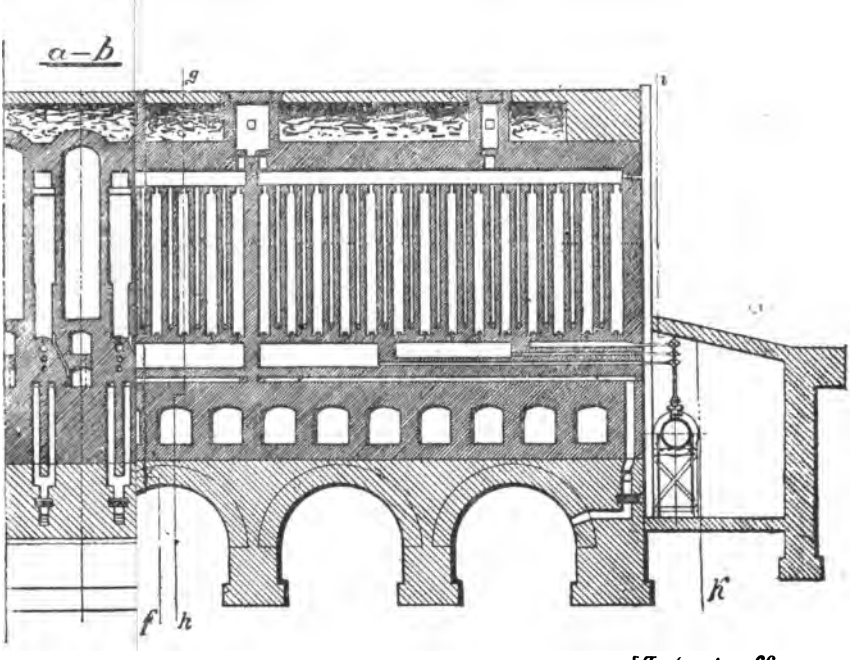
FIG. 53.—BATTERY OF 73 COPPÉE OVENS, CREUSOT WORKS, FRANCE.

FIG. 56.—TWO BATTERIES OF 106 COLLIN OVENS.





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the regenerators. The flame rises, and the waste products pass through the "inner" flues of the bond stones to the regenerators. During the second stage the gas is led into the flues from the upper series of conduits, and the hot air from the regenerators rises through the "inner" flues and meets the gas at the top, the waste products passing down the main vertical flues to the corresponding regenerators. The first series of regenerators is underneath the ovens, and is free of chequer work, the waste gases passing from thence through large regenerators with chequer work before reaching the reversing valve. The regeneration thus takes place away from the oven, the flues underneath the oven not being subjected to any great change of heat. The upper series of conduits is so arranged that the combustion of the gas takes place low enough to act on the charge itself, without unduly heating the crown of the oven. The reversing of the regenerators in some types of ovens causes an alternating variation of temperature from one-half of the oven wall to the other, but in the Collin oven the heating is practically continuous.

CHAPTER IX.

Charging and Discharging of Coke Ovens.

Charging and Discharging Coke Ovens.—The older retort coke ovens were charged in a similar manner to the beehive ovens, by means of tubs, feeding the oven through three or four charging holes in the roof, the charge being afterwards levelled by hand or machinery through levelling holes in the oven doors. These charges of loosely filled slack have now been displaced very largely by compressed charges. This system of compressed charges is a great improvement on the old system. The advantages derived are :—

1. Output increased at least 10 per cent.
2. Denser coke.
3. Amount of coke breeze reduced.
4. Saving in labour.
5. Less wear on oven linings.

The slack after compression is almost half as dense again as in a loose charge, but the time taken to thoroughly coke the denser charge is also longer, and the nett result is a gain in output as stated above. The coke from compressed charges is certainly denser and contains less breeze than that from loose charges using the same quality of slack. The time taken in charging an oven is also reduced, and the smoke nuisance during charging with tubs and levelling, is lessened considerably. As there is a slight clearance between the compressed charge and the oven walls, the slack does not come in contact with the brickwork, which is thus protected from the corrosive fluxing action of the mineral matter in the coal. The following is a general description of the type of compressing plant introduced into England by Mr J. H. Darby. The slack from the washer is first crushed in a Carr disintegrator (shown in Figs. 57, 57*a*).

This consists of two reels A and B mounted on the shafts C and C_1. Each reel consists of two concentric rings in which steel spindles are fixed from 2 to 3 inches apart. The reels are driven by the pulleys D and D_1 in opposite directions and

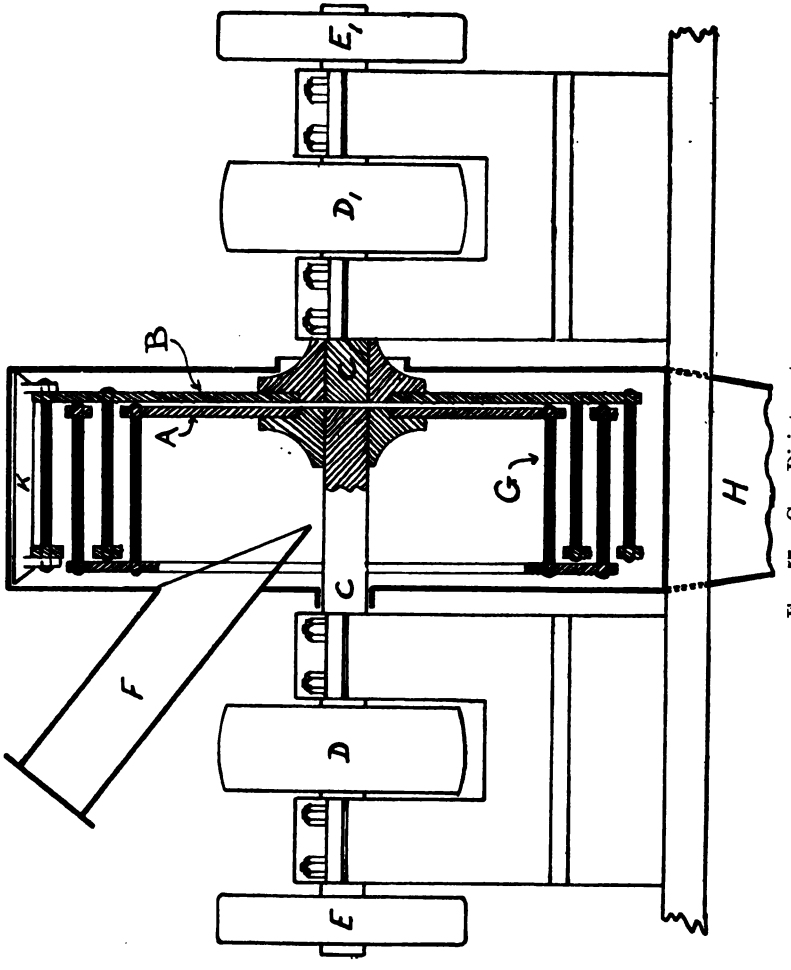

FIG. 57.—Carr Disintegrator.

an even running is secured by flywheels E and E_1 giving the correct balance. The slack is fed from elevators and the shoot F into the centre of the reels, and passes through the bars of reels at G. As these bars are revolving in opposite directions

at a peripheral speed of 6,000 feet per minute, the slack is crushed to a fine state of division and passes down the shoot H. The crushed slack is taken from this shoot to a storage bunker usually situated near the ovens as in Fig. 58. From thence the slack is taken by tubs, or preferably by a conveyor belt A as shown, and delivered into the box B of a charging machine. This box is shaped somewhat similarly to the interior of the coke oven, but the width of the charge can be adjusted by Darby's patent arrangement for closing or opening the sides of

FIG. 57*a*.—One Reel of Carr Disintegrator with Casing removed.

the box by means of the eccentrics D. As the slack is fed into the box it is compressed by the weighted pole F of the stamper E which traverses the whole length of the box. In the system shown, the stamper pole is raised by an eccentric friction wheel driven by electric motor, which also works the travelling gear. The length of "fall" of the stamper pole remains constant, adjusting itself as the charging box is filled. Whilst a single stamper will compress a charge quite satisfactorily, it is an undoubted advantage to have two stamping machines owing

to better compression, saving in time, and the fact that in case of breakdown of one stamper, the other is able to do the work whilst repairs are being conducted.

The drawing shows the charging machine separate and dis-

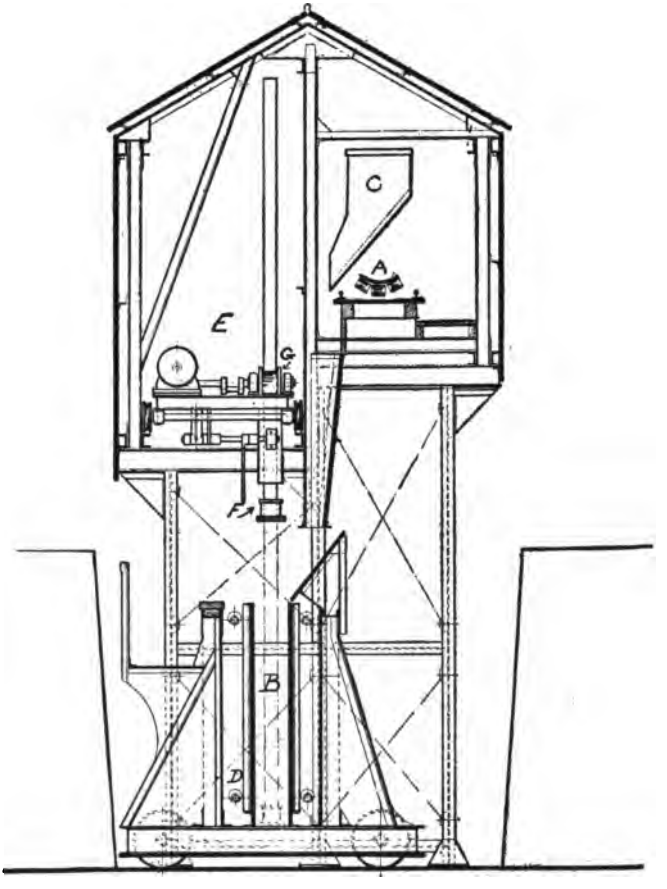

FIG. 58.—Arrangement of Coal Stamping Machinery.

tinct from the ramming machine, but for smaller batteries of ovens it is more economical to use a combined ram and charger, as the lesser number of ovens to be charged per day allows ample time for the charge to be stamped, and then kept waiting until the contents of the oven are discharged by

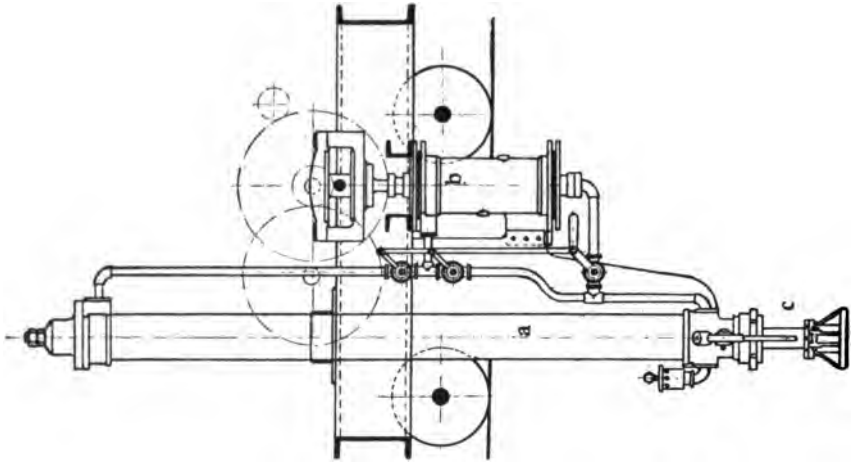

FIG. 58b.—ANOTHER TYPE OF MEGUIN STAMPING MACHINE. (Compressed air.).

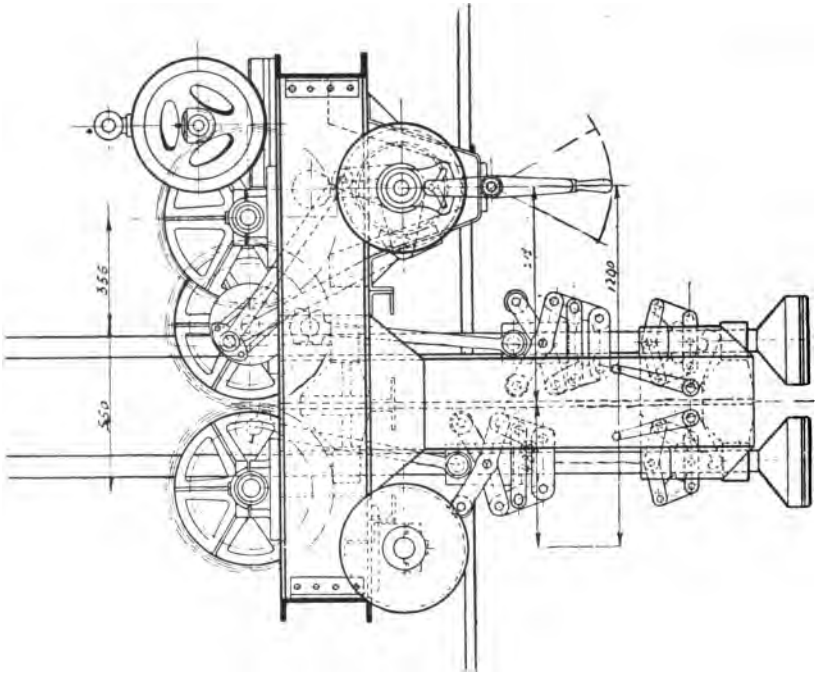

FIG. 58a.—THE MEGUIN STAMPING MACHINE.

the section of the combined machine containing the discharging ram. When the compression of the charge is completed, the

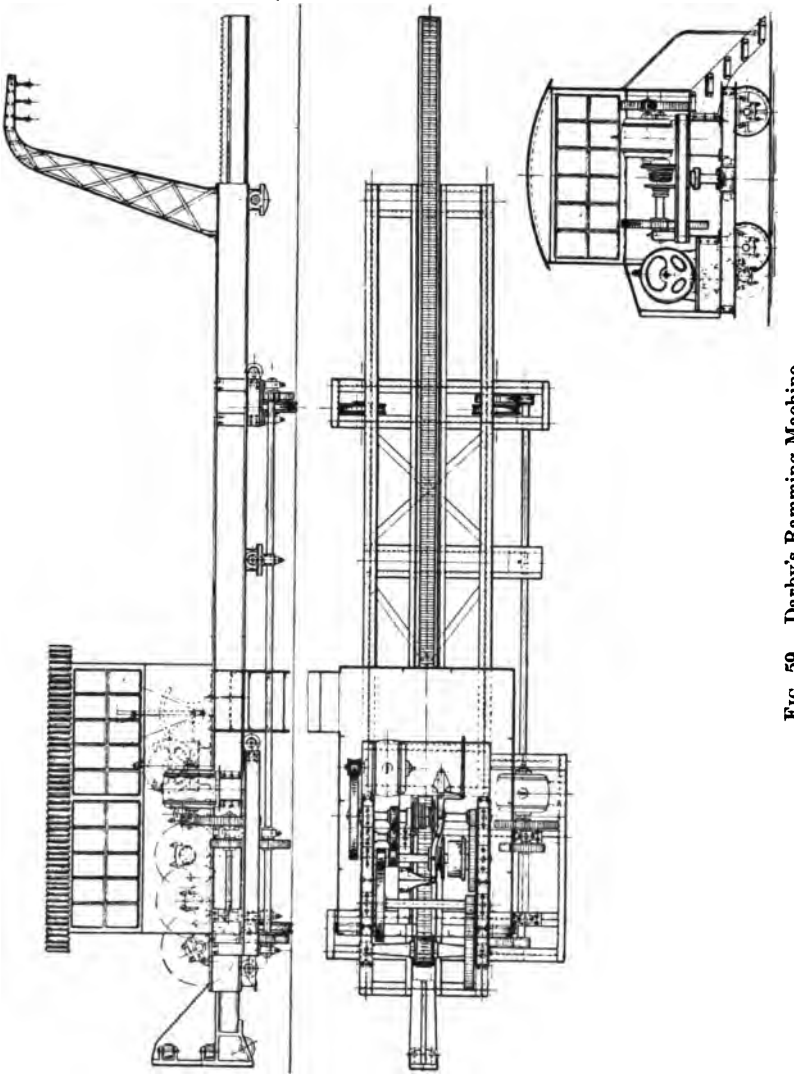

FIG. 59.—Darby's Ramming Machine.

machine travels to the oven requiring a charge, the sides of the "box" are slightly opened by a hand-wheel working the eccentrics D, and the charge is carried into the oven by the peel

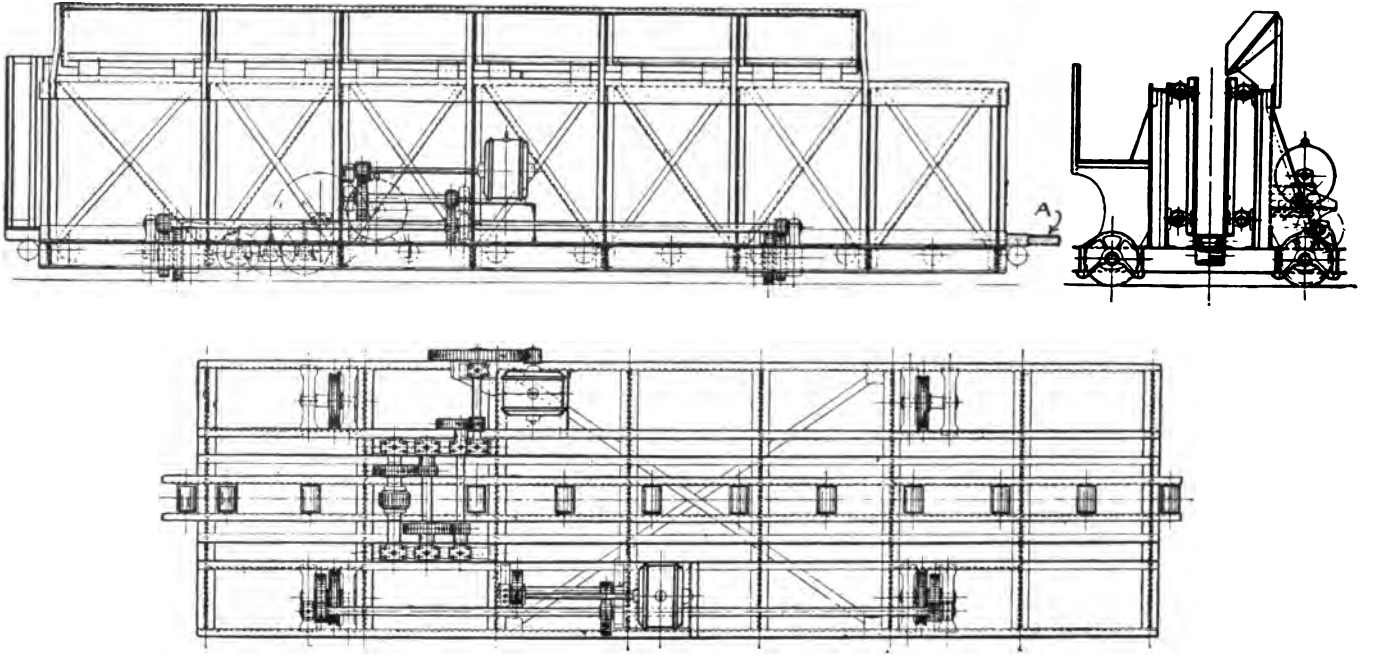

FIG. 60.—DARBY'S COMPRESSING MACHINE.

A (Fig. 60) which forms the bottom of the box. This "peel" is fitted with rackwork underneath into which fits a pinion driven by reduction gear and electric motor. The ramming machine, also designed by Mr J. H. Darby, is shown in Fig. 59. When discharging an oven, the ram travels slowly enough to allow the coke to be thoroughly quenched as described later, but on the return, the speed of the ram is increased about five times in order to avoid unnecessary heating of the ram. This change

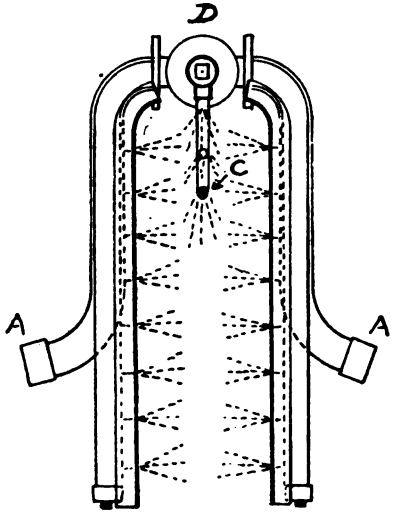

FIG. 61.—Sectional Elevation.

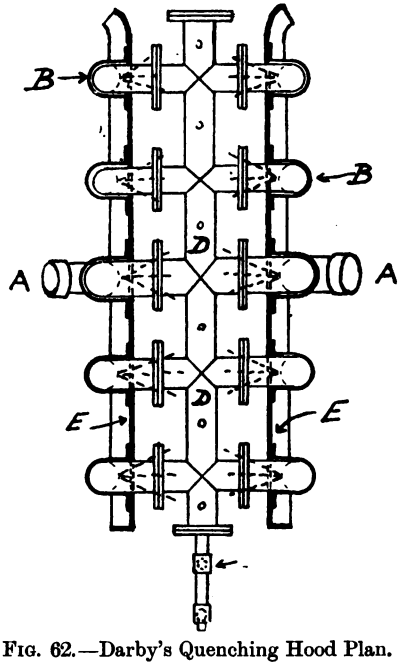

FIG. 62.—Darby's Quenching Hood Plan.

of speed is brought about by clutch gearing without interfering with the speed of the motor.

Another type of stamping machine made by Messrs Fr. Méguin & Co. is shown in Fig. 58*a*. In this type the "pole" is replaced by a steel rod which is raised by a connecting rod worked by a crank. On the upward stroke the rod is gripped by the tong arrangement shown. Towards the end of the stroke the "tongs" come in contact with lugs and are thereby opened, allowing the rod to drop.

Another type, also manufactured by the above firm, is shown in Fig. 58*b*. In this the stamping piston is actuated by the conjoint effect of pressure and vacuum, the stamping cylinder *a* being coupled direct to the compressing cylinder *b*. The speed of this machine is much greater than the former and the capacity increased considerably. The latest type of machine of the above firm has a separate air reservoir, rendering the motion of the compressor absolutely independent of the stamping motion.

The appearance of the coke from a coke plant is a very important matter. Formerly the coke was pushed out by a

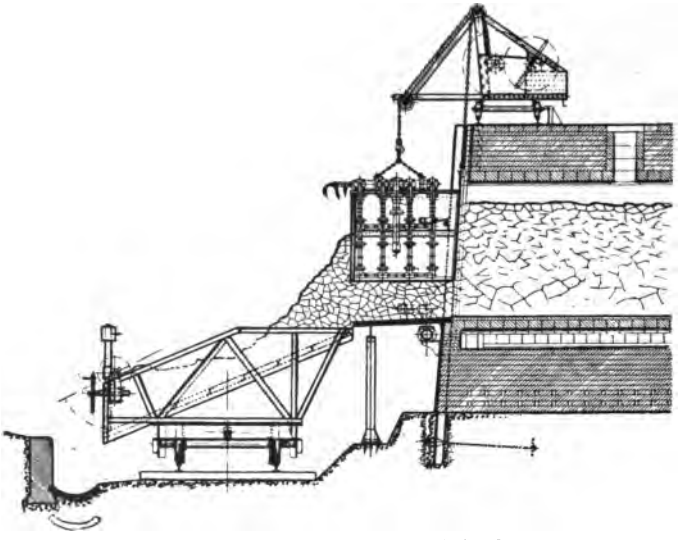

FIG. 63.—Arrangement of Coke Car.

ramming machine on to a horizontal paved floor where it was quenched. The incandescent coke being thus exposed to the atmosphere, suffered through oxidation before being thoroughly quenched, this oxidation giving rise to the dark appearance of the coke. To avoid this, the Semet-Solvay ovens are all fitted with a patent coke quencher (Figs. 61, 62), &c. This quencher is usually carried by the winch which is used for raising the doors on the discharge side of the oven, a small jib being fitted for the purpose. The quencher is placed in front of the oven to be discharged, and is coupled to a water main

FIG. 64.—COKE CAR AT THRISLINGTON COLLIERY.—SEMET-SOLVAY OVENS.

by a flexible hose at A. When the coke begins to emerge, the water is turned on, and passes from A to the branch pipe D, from thence to a series of pipes at B. These pipes are perforated, and the water issues in numerous jets on to the coke which is guided by the steel plating E. Thus the coke is quenched before it really comes in contact with the atmosphere, and a good silvery appearance is obtained. The water supply is usually from overhead tanks to ensure a good pressure.

The coke after leaving the quencher may be dealt with in various ways. Where the coke is to be raised to a higher level and delivered into wagons the patent coke car (Fig. 63) is very serviceable. The car is connected to a hauling rope and is placed opposite any particular oven to receive the charge as it is pushed out by the ramming machine. When the oven is emptied the car is drawn away and the door C is opened, and the contents slide over a screen into wagons. Sometimes the car is dispensed with, and in this case the coke passes down a slope, plated with cast-iron plates, and set at such an inclination as to allow the coke to slide down without undue breakage. The slope delivers the coke on to a plate conveyor, or a flat space at the foot of the slope may be provided to allow the coke to be conveniently forked by hand and loaded into wagons.

CHAPTER X.

Cooling and Condensing Plant, &c.

The bye-products from coke ovens are collected by (*a*) cooling and condensing; (*b*) washing and scrubbing the gas from the ovens. In most cases the slack (usually from a coal washery) contains about 10 per cent. of moisture. On charging an oven, this moisture is expelled as water vapour along with the volatile matter of the coal, and on cooling the gases this vapour is condensed, bringing down with it a portion of the ammonia formed in the oven, the liquid thus forming what is known as ammoniacal liquor. At the same time some of the volatile hydrocarbons are condensed, forming the tar. The mixture of tar and ammoniacal liquor passes to settling tanks, in which the two substances separate, owing to the difference in specific gravity, tar sinking to the bottom and the liquor remaining on top. The gas, in the first place, passes from the oven by means of a stand-pipe (fitted with a valve) into a collecting main. This may be either of the "dry" or the "hydraulic" type. The dry main practically consists of an enlarged tube of steel plating, erected with a definite fall from one end to the other. This main is kept clear by a stream of thin tar or liquor pumped in at the higher end, which washes the pitch and carbon deposits down to the lower end, where they are collected at the sealed overflow.

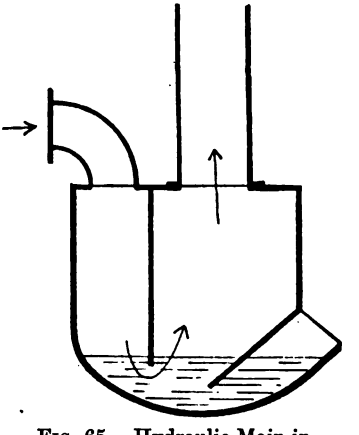

Fig. 65.—Hydraulic Main in Section, showing Seal.

In the "hydraulic" mains the gas is drawn through a "seal"

FIG. 66.—CONDENSING PLANT, CRIGGLESTONE COLLIERY.—OTTO-HILGENSTOCK OVENS.

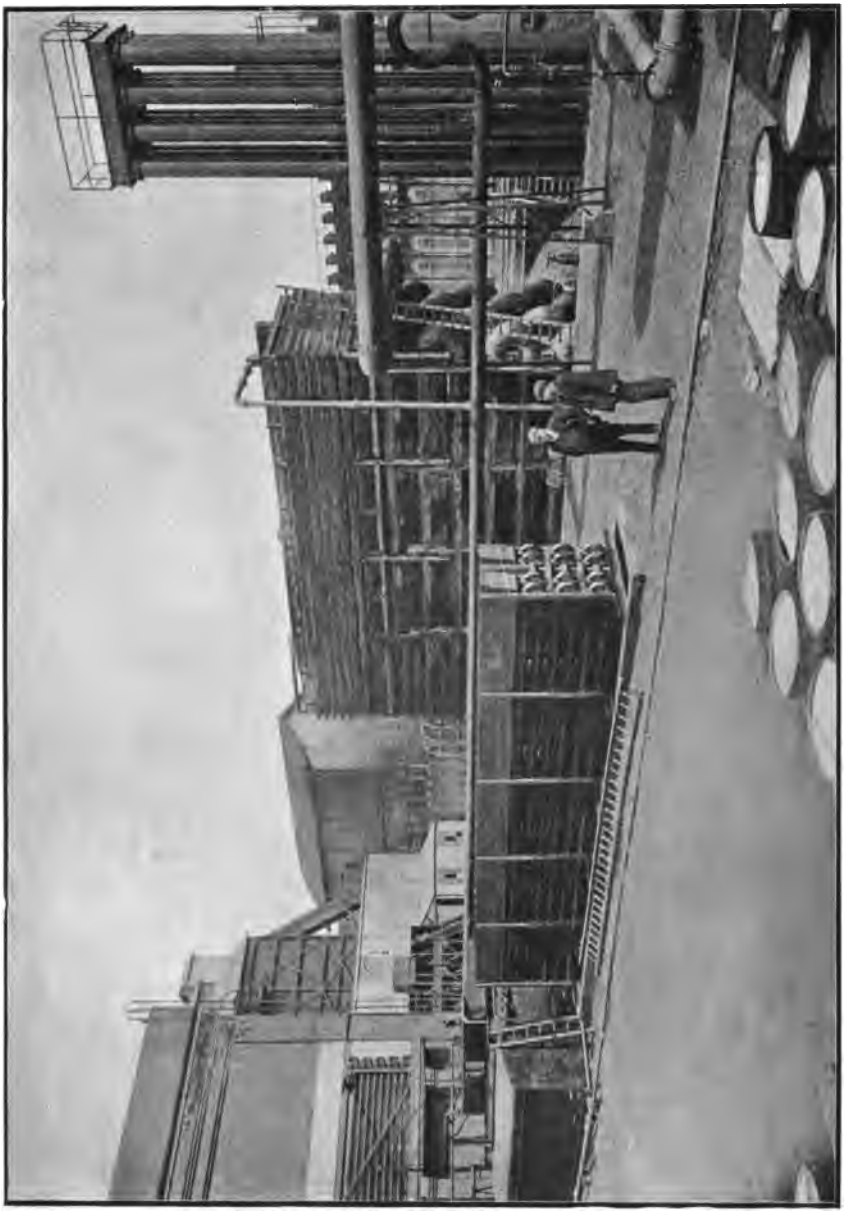

FIG. 67.—COOLING AND CONDENSING PLANT—SIMON CARVES OVENS.

of liquor, thus receiving a preliminary washing and cooling. The ovens may be sealed individually (as in Fig. 19), in which case the stand-pipes are each connected to separate dip pipes, or collectively (Fig. 65), in which case a baffle plate, extending the whole length of the main, and sealed in the liquor, directs the gas through the liquor. The gas from these mains is then conducted through condensers, which may be air-cooled or

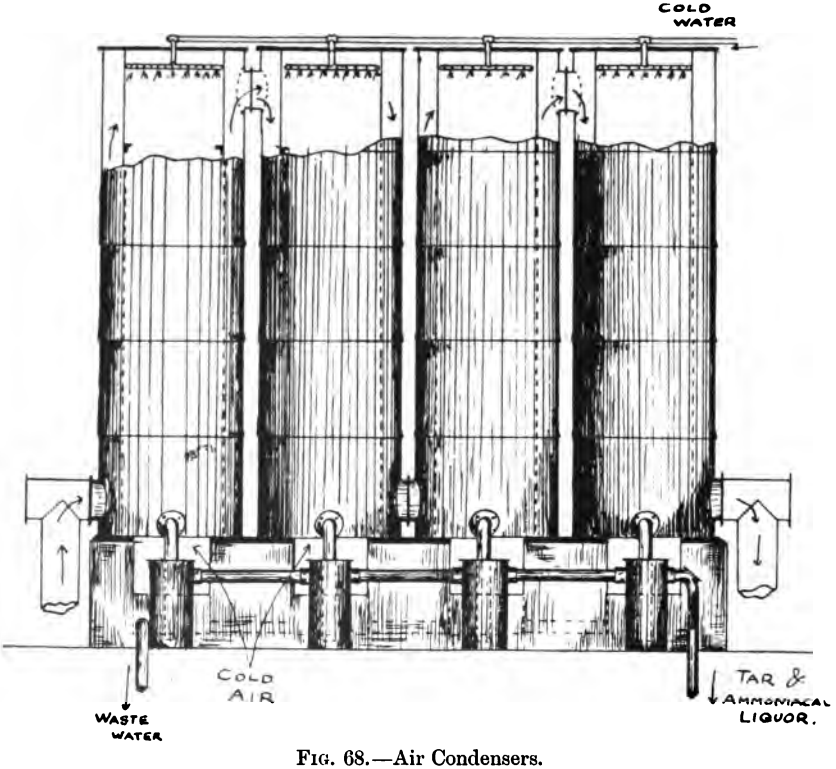

FIG. 68.—Air Condensers.

water-cooled. Some plants use a serpentine arrangement of piping (as at Figs. 66, 67), the gas then passing on to the exhausters and scrubbers, the pipes in some cases being cooled by streams of water playing on them. These pipes are laid with a slight fall, so that the condensed products may be run off at the lowest point through a seal pot. Other plants use air condensers of the type shown in Fig. 68, the principle of

which will be easily seen from the drawing. As air coolers and condensers are, to some extent, dependent on atmospheric conditions, it is advisable to use in conjunction with them water-cooled condensers, of which Fig. 69 is a type. The cooling is

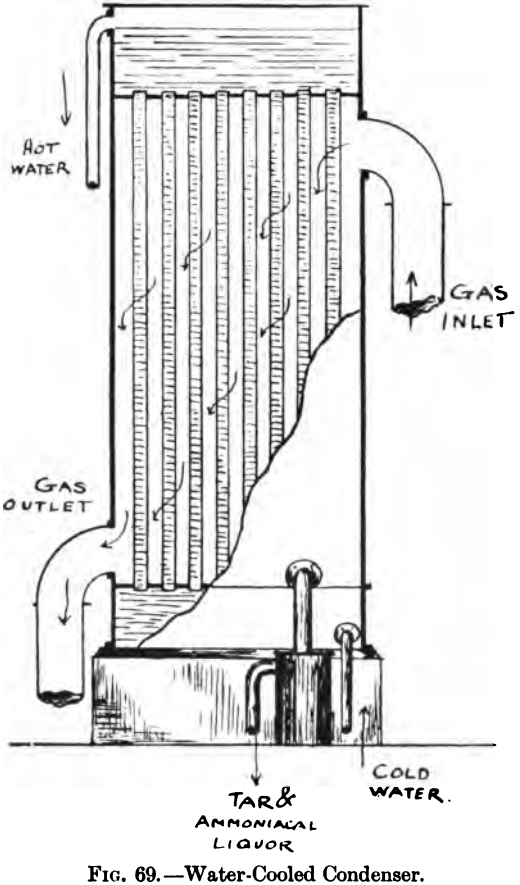

FIG. 69.—Water-Cooled Condenser.

certainly more under control, and uniform cooling may readily be obtained.

Whilst air and water condensers bring down a great proportion of the tar and ammonia from the gas, some of the tar remains in a very fine state of division, and is recovered by means of a washer or a tar extractor. The washer consists of

FIG. 70.—COOLING AND CONDENSING PLANT—SEMET-SOLVAY OVENS. WIGAN COAL AND IRON CO., LTD.

a chamber or chambers, in which the gas is directed through water or ammoniacal liquor by means of a serrated or perforated baffle plate, the depth of seal being sometimes regulated by

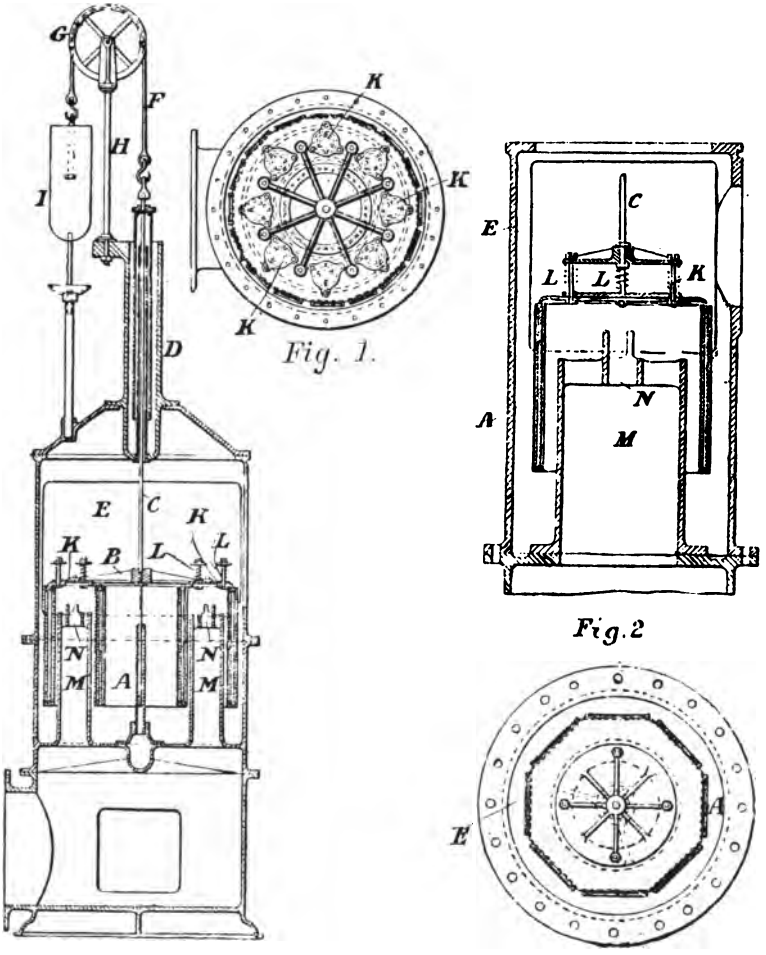

FIG. 71.—Pelouze & Audouin Tar Extractor.

a weir valve. This type of washer also removes some of the ammonia remaining in the gas. For the removal of the tar the apparatus shown in Fig. 71 is also very efficient. It is known as the Pelouze & Audouin tar extractor. In this machine the

gas is broken up into innumerable small jets by perforated plates, each jet impinging on the cool surface of a plate immediately behind the former plate. The impact causes the fine particles of tar to meet together, and the tar accumulating on the impact plate runs off through a sealed overflow. In the type of extractor shown (manufactured by W. C. Holmes & Co., Huddersfield) the gas enters below, and, passing upwards through the annular space M, is directed through the perforated screens A to the upper portion of the apparatus.

FIG. 72.—Condensing Plant, showing Tar Extractor, at Creusot, France.—Coppée Ovens.

To some extent the apparatus also acts as a governor, helping to maintain a steady pressure. The drum to which the screens are attached, is balanced by a counterpoise D, and in case the upper series of perforations becomes clogged with tar or naphthalene, the resistance offered would increase the pressure underneath the drum. This would cause the drum to rise, exposing another row of perforations and relieving the pressure. Thus a steady pressure is kept up. In case the chain holding up the

drum were to break, the above makers have fitted to their machines the relief valves K, which are opened by contact with projections on the gas passages N, the gas then having an absolutely free passage to the outlet.

The appearance of tar is generally well known. It may be described as a dark-coloured, oily-looking liquid, more or less viscous. Its physical nature and chemical composition vary according to the conditions under which it has been produced. It is one of the products of the distillation of coal, both at gas works and also in modern coking practice.

The composition of tar is very complex, and whereas it and ammonia liquor were at one time regarded as residuals having comparatively little value, they are now recognised as valuable bye-products, and one branch of the coal-tar industry alone—that dealing with the dyes—constitutes a most important work.

When tar is heated various products are obtained, according to the temperatures employed. The first effect of heat is to drive off water and ammonia, which, when condensed, constitutes an ammoniacal liquor. On further heating, light oils, such as benzol and naphtha, distil over. Then, as the temperature rises, we obtain in succession the middle oils, such as naphthalene and carbolic acid; then creosote and heavy lubricating oils; afterwards anthracene, and finally pitch, or, if the heating is carried far enough, coke only remains in the retort or distilling vessel. The temperatures of distillation may vary slightly in practice, but a reliable assay of tar may be made by fractional distillation on the following lines:—

Temperatures up to 170° Cent.	- =	Ammonia liquor and light oils.
Temperatures from 170° to 230° Cent.	=	Middle oils.
„ „ 230° to 270° Cent.	=	Heavy oils, creosote, &c.
„ „ 270° to 360° Cent.	=	Anthracene oil.

A fractionating flask with the usual side tube, or an ordinary tubulated retort may be used in the distillation, using 8 or 10 oz. of tar; the several distillates being collected in a graduated measure and their volume noted. The method is especially useful for comparative tests. It is advisable to have a tray filled with sand underneath the retort, then, in

the event of the apparatus breaking, which is always a possible event, there is very little danger of fire, and it certainly helps the mess to be cleaned up more readily. It is preferable to use a thermometer, inserted in the neck of the distilling apparatus to indicate the temperature of the vapour which is distilling over. The changes, however, are very marked in appearance.

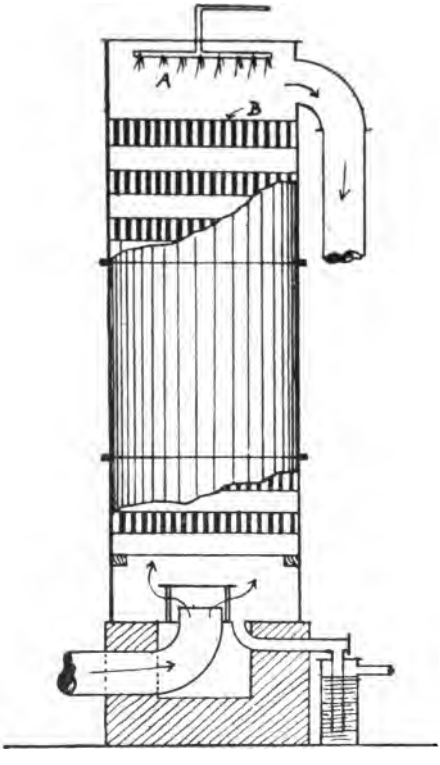

FIG. 73.—Tower Scrubber.

Tar from a coking plant is usually sent direct to the distillers; some works, however, recover light oils. The successful distillation of tar in its entirety is an industry of itself. Water in tar is naturally objected to by distillers, and should not be allowed to exceed 3 or 4 per cent.

The character of the pitch depends upon the temperature at which the distillation was stopped. Soft pitch softens at 40° Cent., medium hard at 60° Cent., and hard pitch at 80° Cent.

Scrubbers.—Whilst the cooling and washing of gases removes the bulk of the ammonia, some small proportion still remains. The last traces of ammonia are not easy to recover, and scrubbers are necessary for this purpose. In these scrubbers the gas is brought into contact with as much wetted surface as possible, the liquid used (either water or weak ammoniacal liquor) being kept as cool as possible. The cooling of the weak liquor, if used, is very important as will be seen from the following table:—

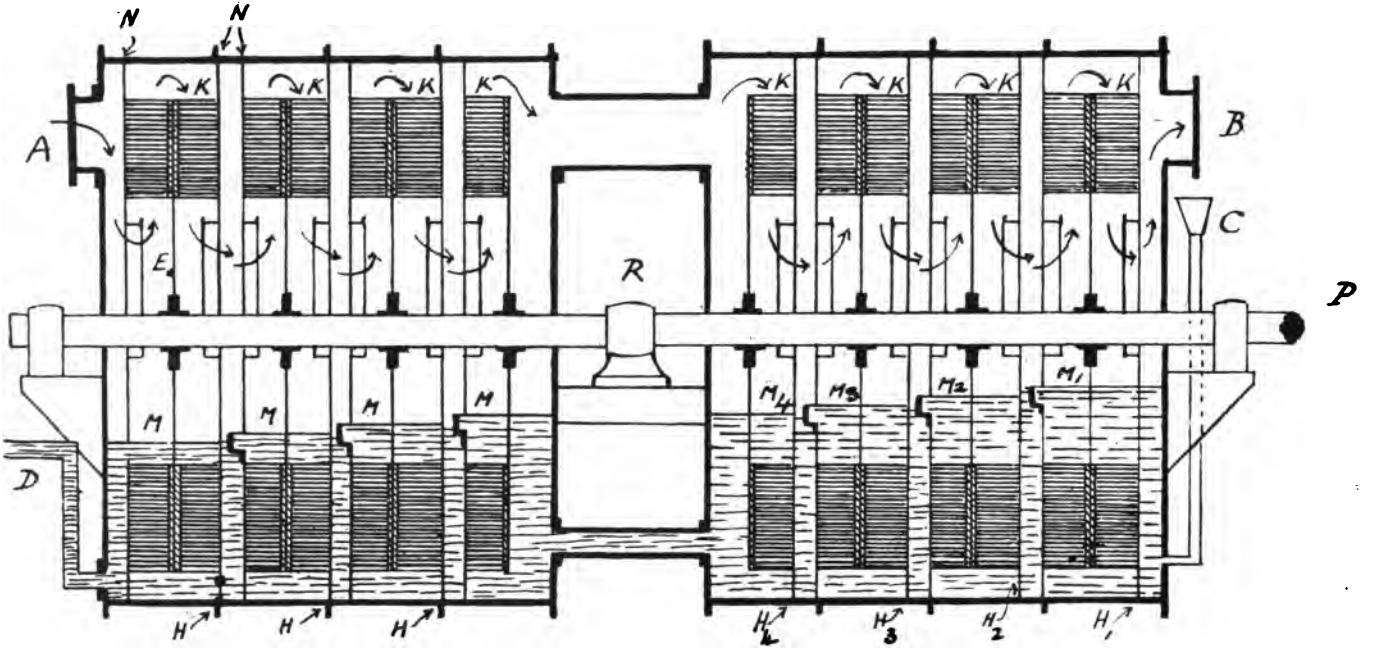

FIG. 74.—REVOLVING WASHER SCRUBBER. (W. C. Holmes & Co.)

Temperature ° Cent.	Grams of Ammonia absorbed by 1 Gram Water.
10	·679
20	·526
30	·403
40	·307

From the above it is evident that the absorbing action of water decreases very rapidly as the temperature rises, and the temperature of the outlet gases of the scrubber should be kept below 30° Cent. The simplest form of scrubber is the tower scrubber. This is simply a tower of steel plating filled either with coke (in pieces of uniform size) or some type of wooden grids as shown in Fig. 73. In this the gas enters at the bottom and passes upwards through a series of boards on edge, each row of boards crossing the preceding one at right angles. The boards are kept wetted by a sprinkler A. The ammoniacal liquor is collected in a seal pot as shown. In some cases these scrubbers are replaced by revolving washer scrubbers of which Fig. 74 is a type. This scrubber, the "New" washer scrubber of W. C. Holmes & Co., Huddersfield, consists of a cast-iron cylindrical chamber laid horizontally. This chamber is divided into sections, as shown. In each section is a circular plate E securely fixed to a common central shaft P. To each side of this plate a series of brushes K is fixed radially, the brushes forming a close fit between the plates N of each section. These brushes, as they revolve, pass through the liquor M in the lower portion of the scrubber, and are thus kept moistened. The weak liquor, or water, is fed into the chamber at C, and leaves the first section by the overflow M_1, passing into the next section through the opening H_2, finally leaving the scrubber through the seal at D. The gas is thus brought into very intimate contact with the washing liquor, and the scrubbing is very efficient. In the later type of scrubber the chamber is divided into two parts as shown, the central bearing being thus quite accessible and clear of the action of the washing liquor. The shaft P revolves at about five revolutions a minute, the gearing being connected to a steam engine or electric motor, the power required being very low.

CHAPTER XI.

Gas Exhausters.

Gas Exhauster.—The gas exhauster is one of the most important machines in connection with modern coke plants. As described in the last chapter, the gas has to pass through various condensers and washers in order to extract the tar and ammonia. These condensers and washers necessarily offer a certain amount of resistance to the gas, and it is the duty of an exhauster to overcome this resistance. The exhauster should be so arranged as to draw off all the gases from the coke ovens without causing either suction or pressure on the oven. If the exhauster were to work too slowly, the gas in the oven would tend to pass into the side flues if any leaks existed in the oven walls, or would pass through the luting of the oven doors. In any case, the bye-products would be lost. If the exhauster were to work too quickly, there would be a tendency to draw air into the oven through the luting of the door, &c. This, besides causing a certain loss in yield of coke and bye-products, lowers considerably the quality of the gas evolved, a matter of great importance where the gas is used for outside purposes. Gas exhausters are of various types, but the types chiefly in vogue at the present time are the rotary exhausters and the jet exhausters.

The rotary exhausters are shown in Figs. 75, &c. Fig. 75 shows one of the earliest type. This is known as a Beale exhauster. It consists of a chamber A, which is stationary. Inside this a drum B, worked by a steam engine or electric motor, revolves, carrying with it a slide C. This slide is carefully fitted in the slot K of the inner drum, and is capable of a sliding movement without allowing gas to pass. As the slide revolves with the drum, it is kept in sliding contact with the inner face of the exhausting chamber, by means of the

block D, which revolves on a pin fixed centrally on the end plate of the exhauster chamber. To ensure gas-tightness and

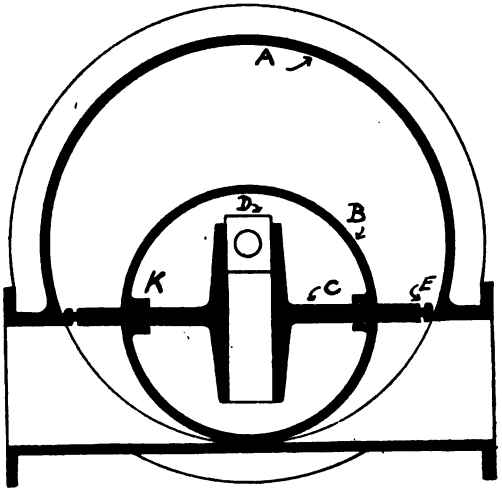

FIG. 75.—The Beale Rotary Exhauster.

to avoid "slip," the ends of the slide are fitted with nose-pieces E which are kept tight against the case by means of springs.

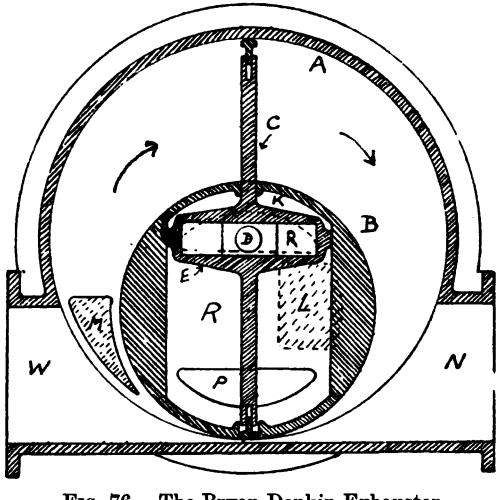

FIG. 76.—The Bryan Donkin Exhauster.

In the modification of this type (Fig. 76) introduced by Mr Bryan Donkin, the motion of the slide inside the drum B is made use of. The drum B is prepared with straight sides between which the portion of the slide marked E is carefully fitted to slide backwards and forwards without slip. At one end of the drum B are openings P and R which, as the drum revolves, pass alternately the port marked L (shaded) in the end casing. In the position shown in the diagram, the space marked K is relatively small, but as the drum revolves in the direction

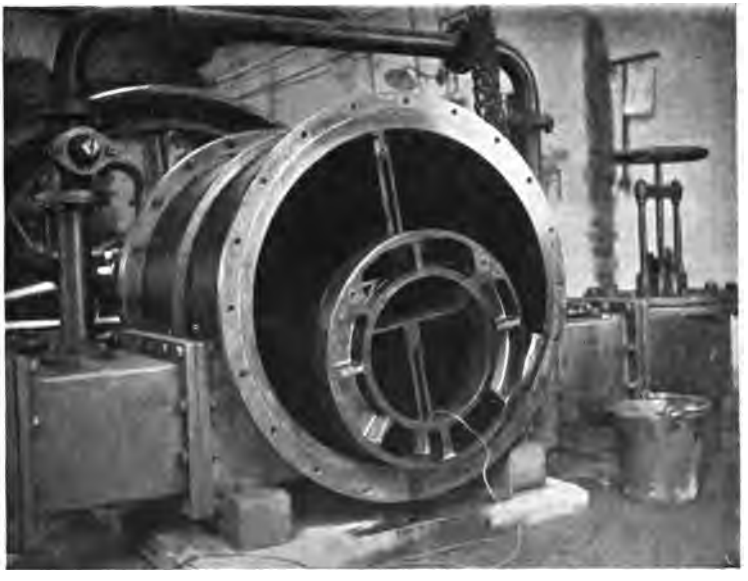

FIG. 77.—The Bryan Donkin Rotary Exhauster. (From a photograph.)

shown, the space becomes larger, creating a suction. As the port R in the end of the drum passes over the port L in the end casing, it draws a portion of gas through L, which is connected to the port M (shaded). Thus the drum is drawing gas from the suction side W during one portion of the revolution. During the second portion, this gas is driven out through ports in the other end casing (not shown on the diagram) to the pressure side N. The capacity of the exhauster is thus increased to the extent of the space represented by the interior of the drum B.

At the same time the number of working parts is increased. Fig. 78 shows a three-bladed exhauster. Owing to the greater number of blades, this type causes less oscillation of the water gauges, where the exhauster has to run slowly. Its action is somewhat similar to the Beale exhauster. A type of exhauster largely used on coke works is the Körtings steam-jet exhauster, shown in Fig. 79. It is on the injector principle, steam being the agent generally used to create the suction. The steam issuing from the nozzle R traverses a series of corresponding nozzles D, C, B of larger areas, thus creating a suction. This

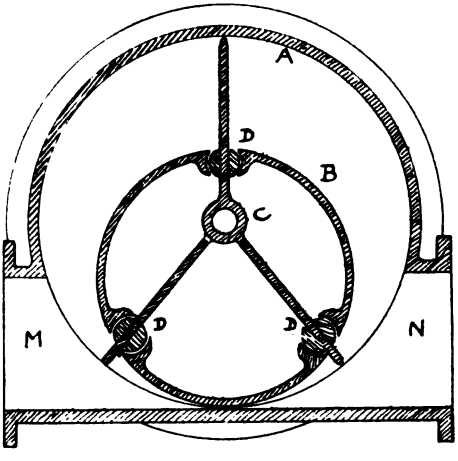

FIG. 78.—Three-bladed Exhauster.

steam is afterwards condensed in water condensers or scrubbers, assisting in the recovery of the ammonia from the gas. The sleeve K, controlled by the screw N, serves to regulate the capacity of the exhauster, whilst the hand-wheel G working the pin-valve, controls to a nicety the amount of steam necessary to maintain the required suction. It may be stated that the amount of steam used is in direct proportion to the amount of gas exhausted. These exhausters, of course, have no moving parts, and occupy a very small space. The exhausters, as used in coke works, are fitted with a plate F to allow easy access to the nozzles for cleaning.

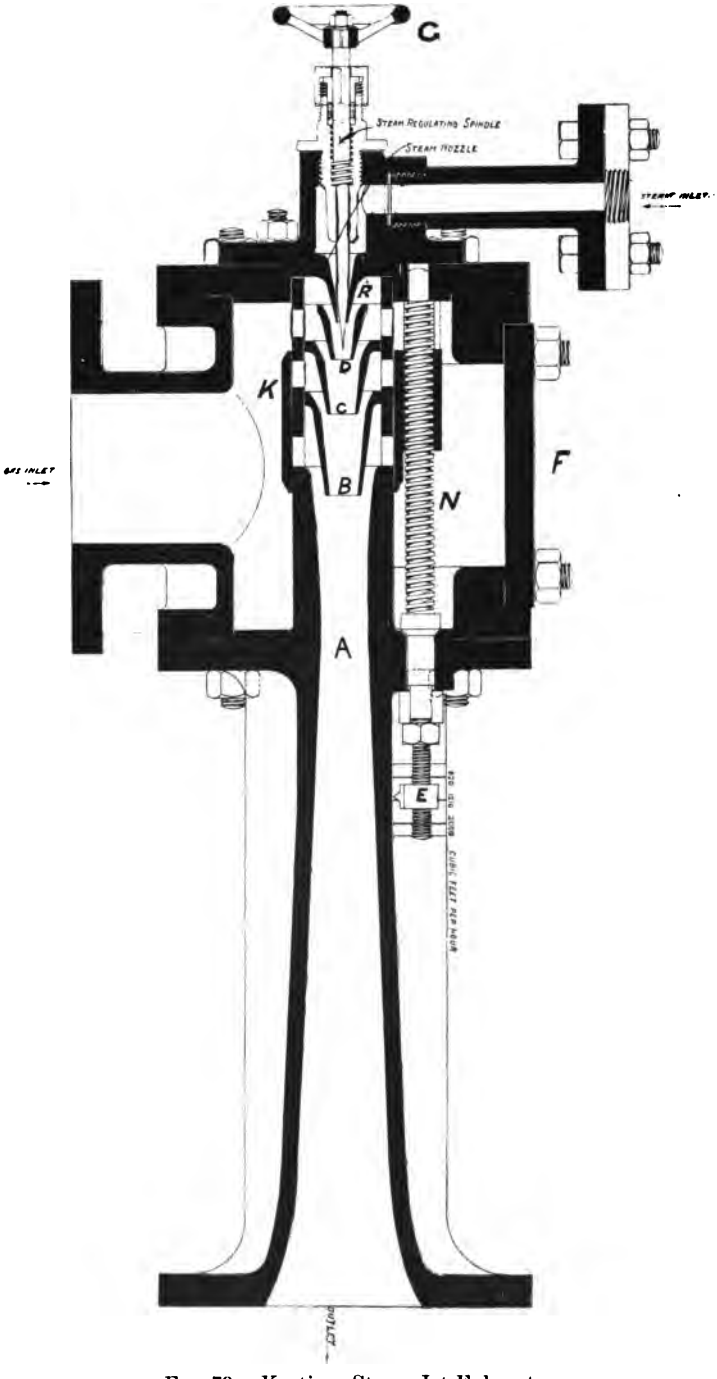

FIG. 79.—Kortings Steam Jet Exhauster.

CHAPTER XII.

Composition and Analysis of Ammoniacal Liquor.

Ammoniacal Liquor.—Ammoniacal liquor, known also as "gas water," is obtained from the hydraulic main, and from the condensers and scrubbers. An approximate analysis of liquor from the various sources is as follows:—

	(Semet-Solvay Ovens.)
From the main - - - - -	·52 % NH_3.
„ condensers - - - -	·48
„ scrubbers - - - -	1·00
„ serpentine washer - - -	1·25

This liquor holds in solution, in addition to free ammonia (ammonium hydrate), various compounds, of which the following are the principal:—

Ammonium carbonates.
„ sulphides.
„ sulphocyanide.
„ sulphate.
„ thiosulphate.
„ sulphite.
„ chloride.
Free hydrocyanic acid.

The specific gravity of the liquor is about 1·05.

The compounds above mentioned are produced in various ways.

Carbon dioxide is always a constituent of the gases coming from the ovens, and is formed by the union of some of the carbon of the coal, with oxygen derived from the air which is unavoidably admitted with the slack during charging. When ammonia gas comes in contact with carbon dioxide, below a certain temperature, one or other of the carbonates of ammonia

is formed according to prevailing conditions. These are readily dissolved in the water which is in contact with the gases in the main and condensers, &c. In fact, all the above-named compounds of ammonia are readily soluble in water. The carbonates are white solids, which, however, cannot exist at high temperatures, and are consequently not formed until below a certain temperature.

The carbonates of ammonia smell of free ammonia under ordinary atmospheric conditions, probably through the action of carbon dioxide from the air, partly due also to the presence of carbamate, since this compound is formed by the interaction of gaseous ammonia and carbon dioxide, if these are dry and not too hot.

Ammonium Sulphide is produced by the action of sulphuretted hydrogen on ammonium hydrate, the former having been derived from the sulphur contained in the iron pyrites always associated more or less with coal. There are several sulphides of ammonium, containing variable amounts of sulphur. It is to the presence of these compounds that the disagreeable smell of the gas liquor is chiefly due. All the sulphides give insoluble precipitates with lead and zinc salts, and on this reaction may be based a method for their estimation. They easily decompose, and are volatile on heating.

The sulphocyanide of ammonium is formed by the union of hydrocyanic acid and ammonium sulphide. It is possible also that carbon disulphide takes part in the formation.

Sulphite of ammonium is produced by the union of sulphur dioxide, derived from the pyrites in the coal, and gaseous ammonia, in a similar manner to the formation of carbonate. The sulphite, in presence of free sulphur, forms hyposulphite or thiosulphate.

Ammonium Chloride is derived from sodium chloride, or common salt, in the coal, and varies considerably with the class of coal used. Some coals contain a fairly large amount of salt, others are practically free. Ammonium chloride, commonly known as "sal ammoniac," is a white solid which readily sublimes, and easily dissociates into ammonia gas, and hydrochloric acid gas.

Free Hydrocyanic Acid is the result of the direct union of carbon and hydrogen with nitrogen at the high temperatures used in coking, so that a portion of the total nitrogen of the coal is thus changed, and not recovered as ammonia.

Treatment of Ammonia Liquor.—Although ammonia liquor is thus shown to be such a complex solution, fortunately all the compounds are amenable to one form of treatment. When the liquor is brought into contact with milk of lime (chemically calcium hydrate), all the ammonia is driven off in the form of free ammonia, or ammonia gas. This reaction is brought about in the "stills" which are fully described in Chapter XIII.

A typical equation for the reaction is as follows:—

$$(NH_4)_2CO_3 + Ca(HO)_2 = CaCO_3 + 2NH_3 + H_2O;$$

or generally expressed—

$$(NH_4)_nX + Ca(HO)_2 = CaX + n(NH_3) + H_2O,$$

where X represents the acid radicle.

As the terms "free" and "fixed" ammonia are often used in speaking of ammonia liquor, it will be well to understand what is implied by them.

The "free" ammonia is that which is given off from the liquor by boiling only, without the addition of an alkali or base, such as soda or lime.

The "fixed" ammonia is that which is not driven off by simply boiling, unless an alkali or base be present in moderate excess. In the "stills" of a recovery plant lime is used. This causes the decomposition of the whole of the ammonium compounds, and the liberation of all the ammonia in the free, gaseous condition, ready for absorption in the sulphuric acid, when it again becomes "fixed" and converted into ammonium sulphate.

Valuation of Ammonia Liquor.—An approximate idea of the strength of ammoniacal liquor may be obtained by means of a Twaddell or other similar hydrometer, but to ascertain the exact amount of ammonia present a chemical test is necessary.

An estimation of the *total* ammonia is usually made, that is

the free and fixed together, and this is done in the following manner:—

Having obtained a representative sample, 10 cubic centimetres are measured off and transferred to a small flask, to the neck of which a rubber stopper has been previously fitted. Through this stopper is a glass tube, bent twice at right angles and connected to a bulbed **U** tube as shown in Fig. 80. Into the bulbed **U** tube is measured 10 c.c. of normal sulphuric acid, that is, of such strength that each cubic centimetre contains ·049 gram of sulphuric acid or 49 grams per litre. A moderate excess of a strong solution of sodium hydrate (caustic soda) is now added to the flask, and the stopper quickly replaced, gentle heat being then applied to the contents of the flask, gradually increasing it so as to maintain a gentle boil for about half an hour. By this means the whole of the ammonia is distilled over into the acid with which it combines, forming ammonium sulphate, and neutralising a proportional amount of the free acid. It then remains to carefully detach the **U** tube and its contents, and transfer the solution to a glass beaker or porcelain basin, the latter preferred, rinsing out the tube with distilled water several times, care being taken to avoid any loss by splashing, &c. It is now necessary to estimate how much of the acid has been neutralised by the ammonia which has passed into it. This is done by adding two or three drops of a solution of methyl orange to the liquid in the basin and then adding carefully a normal solution of sodium carbonate or hydrate until the pink colour of the solution is changed to yellow. The end reaction is very distinct and indicates exactly when the remaining excess of acid has become neutralised by the standard solution of soda. Each cubic centimetre of normal sulphuric acid neutralised by the ammonia which has been liberated from the 10 c.c. of ammoniacal liquor, represents ·017 gram of ammonia (NH_3).

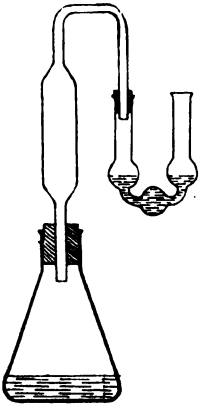

FIG. 80.—Apparatus for the Estimation of Ammonia.

An example of the figures in an actual test will no doubt make the process more comprehensible.

10 c.c. $\frac{N}{H_2SO_4}$ originally used in U tube.

3 c.c. $\frac{N}{NaHO}$ required to neutralise after distillation is complete.

$10 - 3 = 7$ c.c. $\frac{N}{H_2SO_4}$ neutralised by the ammonia from 10 c.c. of ammonia liquor,

and each c.c. sulphuric acid neutralised = ·017 gram ammonia (NH_3).

$\therefore$ $7 \times ·017 = ·119$ gram ammonia from 10 c.c. of ammonia liquor,

and hence 1·19 per cent. ammonia (NH_3).

If it is desired to estimate the free ammonia and the fixed ammonia in a liquor, separately, a measured quantity is taken as before, and distilled over into a known volume of standard acid, *without adding sodium hydrate* to the solution in the flask. On titration with standard soda as before, the figure obtained gives the free ammonia only. This amount deducted from the total ammonia, as determined by a separate test, represents the ammonia in the fixed condition.

Concentrated Liquors.—In many plants, instead of recovering the ammonia in the form of sulphate, it is concentrated in the form of liquor and sold as such. The strength of such liquor varies from 15 to 20 per cent.

The liquor may be tested by the hydrometer as to its strength, but it is preferable again to use a chemical method. It is found that with fresh liquor it is sufficiently accurate to titrate direct with normal sulphuric acid, that is without having to distil with caustic soda. If, however, the liquor is a few days old it is necessary to distil with soda into normal acid as described in the testing of ammoniacal liquor, otherwise too low a result is obtained. In dealing with a liquor so strong as concentrated liquor usually is, it is either necessary to determine its specific gravity by means of the specific gravity bottle (see page 22), or to actually weigh the liquor used in the estimation. This latter is preferable, and may be carried out as follows. Weigh a small conical glass flask containing a few cubic centimetres of distilled water, then add *about* 5 c.c. of the con-

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centrated liquor whose strength is to be determined, and weigh again, having stoppered the flask up with a cork and rubber tubing on which there is a spring clip. By this means, the *weight* of the liquor used is ascertained. This is then distilled with excess of caustic soda into about 50 or 60 c.c. of normal sulphuric acid, carefully measured of course. Gradually warm up the flask and contents and distil at a brisk boil for about twenty minutes. Each cubic centimetre of normal sulphuric acid neutralised represents ·017 gram of ammonia (NH_3). It may be explained that when dealing with an ordinary ammoniacal liquor, say of 1·0 to 1·5 per cent. strength, it is sufficiently accurate to *measure* the 5 or 10 c.c. taken for analysis, and to take it that this volume weighs 5 or 10 grams, as the case may be. But in a highly concentrated liquor containing from 16 to 20 per cent. of ammonia it would be misleading to do this and consequently it is necessary to weigh the portion taken, or to determine the specific gravity of the liquor and from that calculate the weight of any measured volume dealt with.

Estimation of Total Sulphide in Liquor.—The principle of this estimation is based upon the fact that when a solution of zinc chloride (ammoniacal) is added to the ammonia liquor the sulphides are precipitated as zinc sulphide. The solution is boiled up, and after settling, the precipitate is filtered off and washed several times with hot water, and finally titrated with a standard solution of iodine, decinormal strength, whereby the amount of sulphur in the form of sulphide is readily calculated. Every 97 parts of zinc sulphide are equivalent to 32 parts of sulphur or 34 parts of sulphuretted hydrogen.

An example of this method is given in the Chief Inspector's Forty-second Report on Alkali Works, to the Local Government Board.

Estimation of Sulphuretted Hydrogen in Liquor.—This estimation is made by boiling a measured volume of the liquor and absorbing the sulphuretted hydrogen which is thus driven off, in a solution of either a lead, copper, or cadmium salt, &c. The metal is precipitated as sulphide, and may be then, after filtration and washing, titrated with a standard solution of

TO AMMONIA

iodine, or may be oxidised by means of bromine and precipitated in the form of barium sulphate—compare method for estimation of sulphur in coal and coke, page 25.

The authors have used an ammoniacal solution of cadmium nitrate, and find the precipitate of sulphide thus obtained very workable, and can be titrated with iodine, in acid solution, or oxidised to sulphate as described, giving accurate results.

The same apparatus may be used as that in which the valuation of ammonia liquor for ammonia is carried out. (See sketch.) 10 c.c. of the liquor is usually sufficient to work upon.

Estimation of Carbon Dioxide.—Portion of the ammonia in ammoniacal liquor exists in the form of carbonate, and this may be estimated by making use of the following reaction:—

$$CaCl_2 + (NH_4)_2CO_3 = CaCO_3 + 2NH_4Cl.$$

That is, by adding a solution of calcium chloride to the liquor, the carbonate of ammonia becomes changed into the insoluble carbonate of lime, which may then be separated by filtration, washed, dried and ignited to oxide, or titrated with standard hydrochloric acid, when

$$CaCO_3 + 2HCl = CaCl_2 + H_2O + CO_2,$$

which denotes that every 100 parts by weight of calcium carbonate will neutralise exactly 73 parts by weight of hydrochloric acid, and consequently by estimating how much acid is neutralised by titration, the amount of calcium carbonate is determined. Every 100 parts of calcium carbonate are equivalent to 44 parts of carbon dioxide.

If the carbonate be ignited to oxide of calcium and the latter weighed, every 56 parts by weight represent 44 parts of carbon dioxide.

Both methods give accurate results. The volumetric one is well exemplified in the Chief Inspector's Report on Alkali Works for the year 1905.

Estimation of Hydrocyanic Acid in Liquor.—This is done by distilling a measured quantity, 50 c.c. usually, into a solution of caustic soda of semi-normal strength—20 grams per litre.

It is necessary to add a moderate excess of lead nitrate

solution before distilling, to the liquor in the flask, the object being to retain the sulphuretted hydrogen, in the form of lead sulphide, which would otherwise interfere with the titration of the distillate. The hydrocyanic acid becomes converted into sodium cyanide. It remains now to add to this solution a few crystals of potassium iodide as an indicator, and to run in a measured quantity of standard silver nitrate solution until the liquid becomes just cloudy or opalescent. This indicates that all the cyanide present has become converted into the double cyanide of sodium and silver, and that silver iodide is beginning to form, causing the opalescence. Each cubic centimetre of decinormal silver nitrate used represents ·0054 gram hydrocyanic acid.

Typical examples of tests of ammoniacal liquor for carbonic acid, hydrocyanic acid, ferrocyanide and thiocyanate, sulphide, thiosulphate and sulphite, sulphate, total sulphur and chloride are given in the Report of the Chief Inspector on Alkali Works to the Local Government Board for 1905. In this and other Reports to the Local Government Board will be found the results of a very considerable amount of time and care, which have been expended by Mr R. Forbes Carpenter and Mr Linder, in investigating and developing the best and most reliable methods for the analysis of ammoniacal liquors.

CHAPTER XIII.

Working up of Ammoniacal Liquor.

Working up of Ammoniacal Liquor.—The ammoniacal liquor from coke works may be dealt with in two ways. It may be converted into sulphate of ammonia by combination (under certain conditions) with sulphuric acid, or by a simple process of distillation and condensation may be converted into concentrated ammoniacal liquor. In the former method the principle may be briefly described as follows:—The ammoniacal liquor

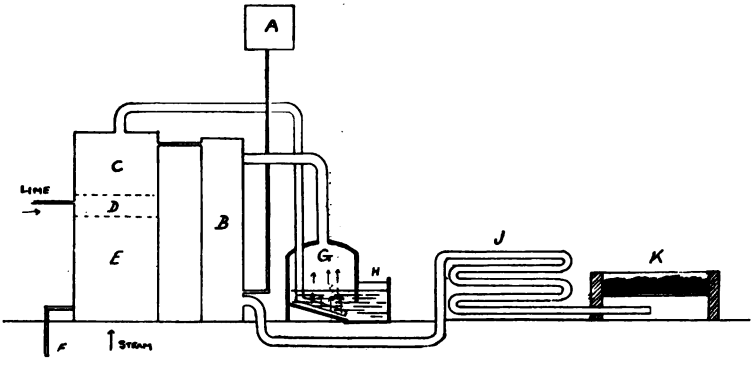


FIG. 81.—General Type, Sulphate of Ammonia Plant.

is brought into contact with live steam and milk of lime. The free ammonia (see page 98) is expelled by the action of the steam alone, but the fixed ammonia requires the presence of a stronger base, such as lime, for its expulsion. The ammonia passes forward with this steam, and bubbles through a bath of sulphuric acid. The ammonia combines with the sulphuric acid (the chemical reaction giving off considerable heat), $2NH_3 + H_2SO_4 = (NH_4)_2SO_4$, and the steam passes through, being

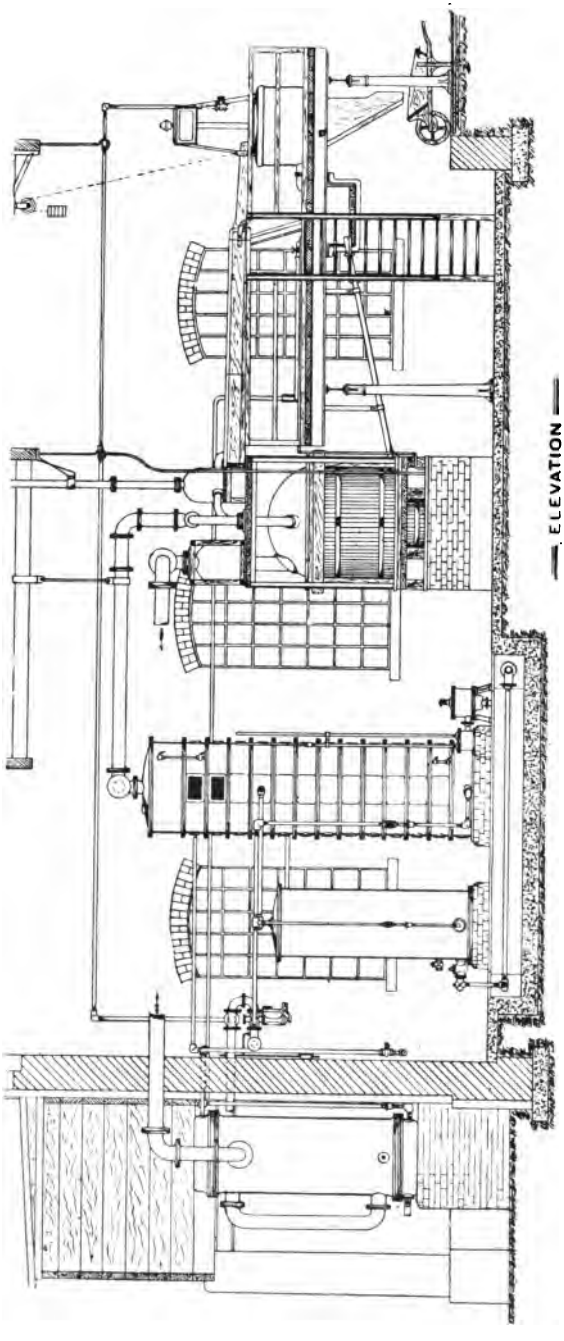


FIG. 81a.—SULPHATE OF AMMONIA PLANT. (Chemical Engineering Co.)

condensed in some form of cooler. The sulphuric acid ultimately becomes neutralised by the ammonia, and crystals of sulphate of ammonia are formed. These crystals are collected by various means, and, after being dried, form the sulphate of ammonia of commerce. The general type of plant is shown in Fig. 81. C, D, E is the ammonia still. The liquor is fed into the still at the top, after a preliminary heating by the waste gases of the saturator G. This preliminary heating is brought about in the superheater B, usually of the multitubular type, the liquor entering at the bottom and leaving the top at a temperature of about 90° Cent. The heated liquor passes on to the still, which is divided into a series of compartments, as shown in Fig. 82 (a section of a "Wilton" still, by the Chemical Engineering Co.). The liquor overflows from one compartment to another,

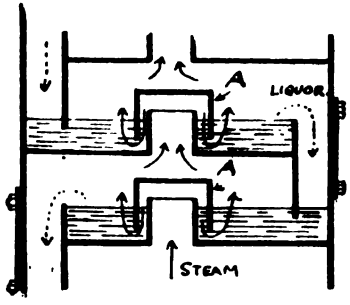


FIG. 82.—"Wilton" Still.

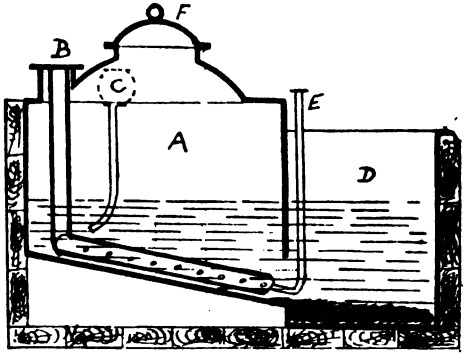


FIG. 83.—Saturator.

taking the course shown by the dotted arrows. Steam enters at the bottom of the still, and is directed through the liquor in the several compartments by the hoods A, taking the direction shown by the plain arrows. The edges of the hoods are serrated to obtain an intimate contact between the steam

and the liquor. About half-way down the still, the milk of lime is added, a compartment being deepened in some cases, as in Fig. 81, to ensure an efficient mixing of the lime and ammoniacal liquor, or preferably the mixing may be done in a separate vessel, as in Fig. 81, A, the liquor in the latter case passing from the upper portion to the mixing chamber and back to the lower portion of the still. In either case the liquor,

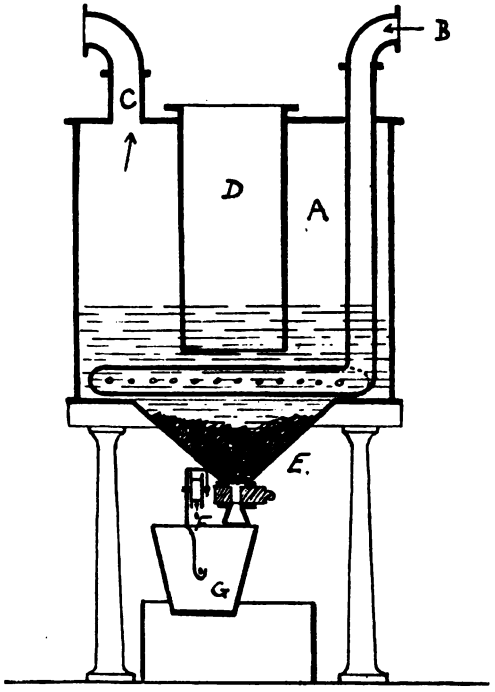


FIG. 84.—Self-Discharging Saturator.

mixed with milk of lime, is deprived of the whole of its ammonia, and leaves the still at the bottom as "waste liquor." The waste liquor passes through a seal pipe or regulator in order to maintain a sufficient pressure of steam, and the solid matter is deposited in settling pools. The ammonia vapour and steam are conducted to a "saturator," in which the ammonia is absorbed. The various types of saturator are shown in Figs. 83, 84, and 85, and the first type (Fig. 83) is a sketch of a

FIG. 85.—SATURATOR. (Joseph Taylor & Co.)

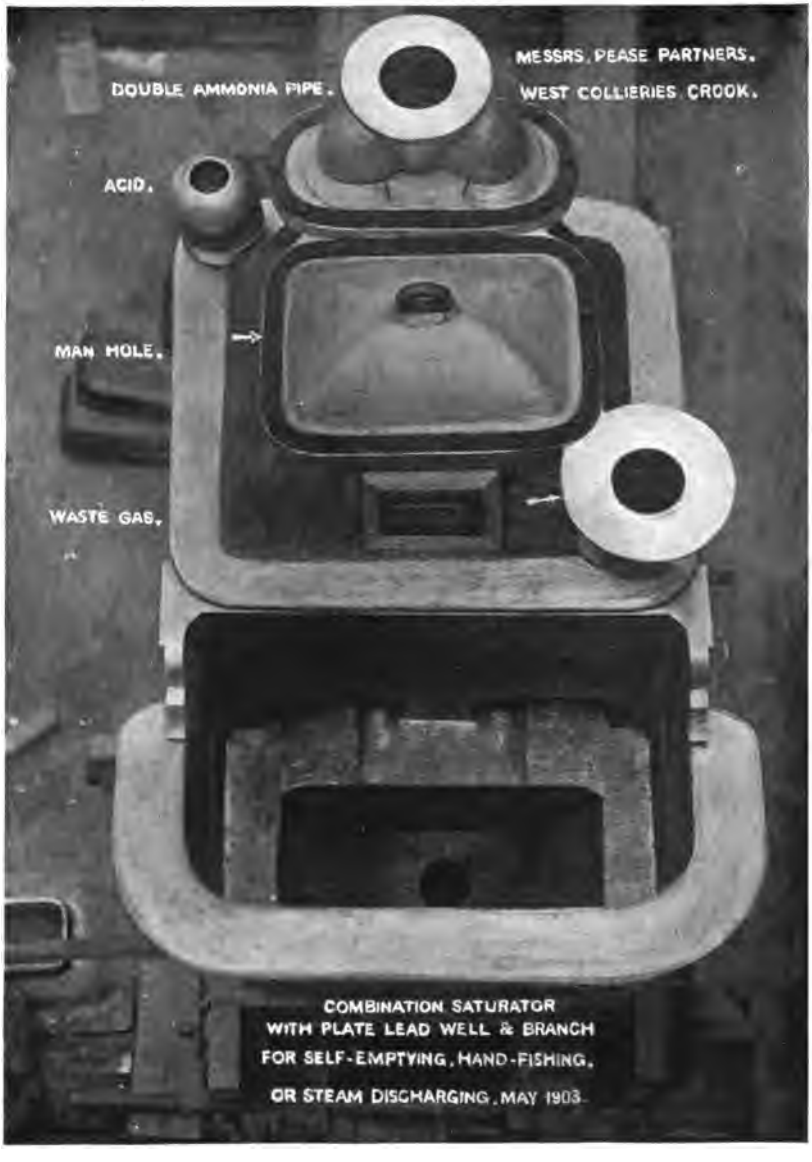


FIG. 86.—SATURATOR. (Joseph Taylor & Co.)

saturator made by Joseph Taylor & Co., of Bolton. This is of a simple but substantial type, being made of solid plate lead. The ammonia vapour is conducted by means of a "blow-pipe" B, consisting of a perforated lead pipe, through the acid in the chamber A. The crystals, as they are formed, fall to the fishing well D, and are collected by perforated copper scoops.

The blow-pipe is kept clear of sulphate by steam from a small perforated pipe E, which blows the sulphate towards the well. In the larger sizes of saturators in this type the sulphate

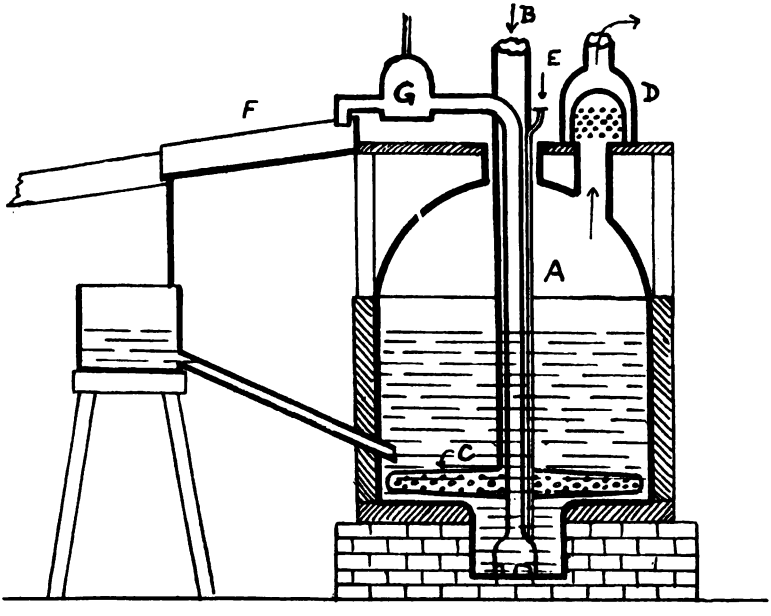


FIG. 87.—"Wilton" Saturator.

is raised by a "Wilton" discharger, similar to the one shown in Fig. 87. The saturator shown in Fig. 84 is of the self-discharging type, and is well adapted for larger outputs. The chamber A is usually of cast iron, lead lined. The blow-pipe is shown at B, and the condition of the "boil" of the saturator may be seen through the lead seal D. The sulphate, as formed, is run off by means of the valve at E on to a draining table direct, or into the travelling skips G. The "Wilton" saturator (Fig. 87) is of the enclosed type. The principle can be easily understood from

the drawing. The sulphate in this case is elevated by the discharger on the ejector principle, the steam inlet being shown at E. The sulphate from this saturator is usually run in a creamy consistency direct into a "Wilton" centrifugal drying machine (Fig. 88). This consists of a basket made of perforated copper and attached to the spindle K. The basket, after being filled with the wet sulphate, is driven at considerable speed by a steam engine or electric motor. The liquor is thus driven by centrifugal force to the perforated sides of the basket

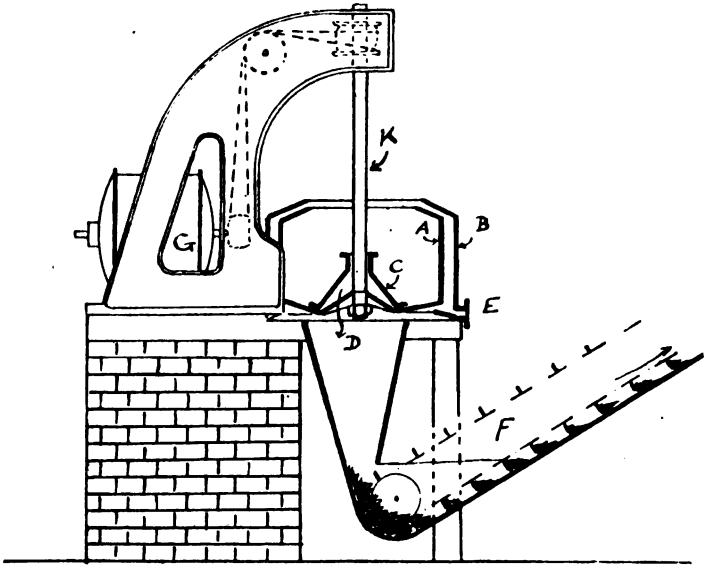


FIG. 88.—Centrifugal Drying Machine.

and passes through. This liquor is then caught by a lead casing B, flowing out at E, either to a mother liquor well or, more usually, back into the saturator. The sulphate is left in the basket in a very dry state, and on lifting the valve C (loose on the spindle) passes through openings D into a shoot. From this it may be wheeled into the storage bins, or carried by elevators of the scraper type shown at F.

The working of the saturators is as follows:—The bath is first made up of a mixture of sulphuric acid and water, in such proportion as to show a specific gravity of 72° Twaddell.

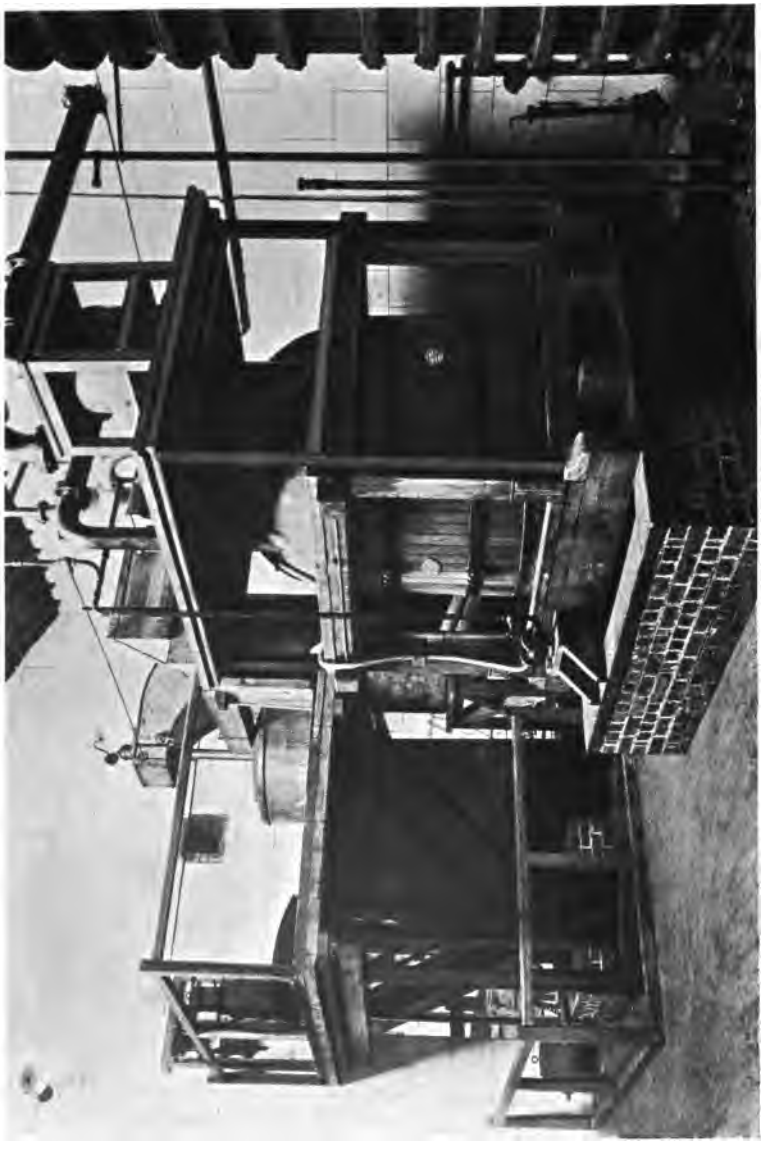


FIG. 89.—SULPHATE PLANT, BARGOED COLLIERY—KOPPERS OVENS.

After the first bath mother liquor is used instead of water, or along with it. As the acid in the saturator takes up the ammonia, the reaction tends to keep up a good "boil." A good boil is really essential in the making of salt of satisfactory quality, and sufficient steam should be kept on the ammonia still to keep the superheater fairly warm. At the same time the overflows of the still must be kept clear to prevent priming or carrying over of the liquor into the saturator. As the process of absorption continues, the specific gravity of the bath becomes lower, and at about 60° Twaddell crystals begin to form, and shortly afterwards, the sulphate must be fished out or run off. If the bath were kept in this condition for any length of time, the acid would become completely neutralised, losing the power of absorption, thus giving rise to loss of ammonia as well as discoloured sulphate. As soon as the bulk of sulphate has been extracted, the bath is brought up to the original strength with fresh acid and mother liquor. The waste gases from the saturator consist of steam, sulphuretted hydrogen, carbon dioxide, and cyanogen. This steam is condensed in the superheater and in condensers (either serpentine or of the multitubular type), and brings down with it a portion of these noxious gases. This liquor, appropriately called "devil liquor," is usually pumped back into the still. An analysis of this liquor gave:—

Ammonia - - - -	·068	**gram per**	**100 c.c.**
Free hydrocyanic acid -	·016	„	„
Fixed hydrocyanic acid -	·86	„	„
Free sulphuretted hydrogen	·0053	„	„
Total sulphides - - -	4·78	„	„

Most of the sulphuretted hydrogen, carbon dioxide, and hydrocyanic acid pass on and form a source of danger in the atmosphere, unless efficiently treated in some way. An approximate composition of the gas is:—

Sulphuretted hydrogen -	2·0	**per cent. by**	**volume.**
Hydrocyanic acid - -	2·0	„	„
Carbon dioxide - - -	82·0	„	„
Nitrogen - - - -	14·0	„	„

Should the saturators not be working efficiently from any cause, some ammonia will escape them and be contained in this waste gas, which means loss and contamination of the sulphur if recovered in a Claus plant.

CHAPTER XIV.

Treatment of Waste Gases from Sulphate Plants.

Sulphuretted hydrogen gas may be dealt with in several ways. At some works it is burnt, and the sulphur dioxide thus produced, used for the manufacture of vitriol. At others, the gases are passed through purifiers containing layers of bog ore or oxide of iron, alone or mixed with sawdust, which takes up the sulphur and some of the cyanide. The sulphuretted hydrogen in passing through this material is decomposed, its sulphur being taken up by the iron oxide forming sulphide, thus:—

$$\underset{\text{Bog Ore}}{Fe_2O_3,H_2O} + \underset{\substack{\text{Sulphuretted}\\\text{Hydrogen}}}{3H_2S} = \underset{\substack{\text{Ferric}\\\text{Sulphide}}}{Fe_2S_3} + \underset{\text{Water.}}{4H_2O.}$$

Also,

$$\underset{\text{Bog Ore}}{Fe_2O_3,H_2O} + \underset{\substack{\text{Sulphuretted}\\\text{Hydrogen}}}{3H_2S} = \underset{\substack{\text{Ferrous}\\\text{Sulphide}}}{2FeS} + \underset{\substack{\text{Free}\\\text{Sulphur}}}{S} + \underset{\text{Water.}}{4H_2O.}$$

When the bog ore becomes highly charged with sulphide it is exposed to the action of the atmosphere to "revivify" it. This comes about by the action of oxygen, thus:—

$$\underset{\substack{\text{Ferric}\\\text{Sulphide}}}{Fe_2S_3} + \underset{\text{Air}}{3O} = \underset{\substack{\text{Ferric}\\\text{Oxide}}}{Fe_2O_3} + \underset{\substack{\text{Free}\\\text{Sulphur.}}}{3S.}$$

Also,

$$\underset{\substack{\text{Ferrous}\\\text{Sulphide}}}{2FeS} + \underset{\text{Air}}{3O} = \underset{\substack{\text{Ferric}\\\text{Oxide}}}{Fe_2O_3} + \underset{\substack{\text{Free}\\\text{Sulphur.}}}{2S.}$$

This revivified material, consisting of ferric oxide and free sulphur, is used over and over again in the purifiers until it becomes charged with 60 to 70 per cent. of sulphur, when it is disposed of generally to vitriol manufacturers for the production of sulphuric acid.

The problem of dealing with the waste gas comes under the control of His Majesty's Inspectors under the Alkali Act, and it was acting upon the instructions and experience of Mr Herbert Porter, the district inspector, that a Claus plant was erected as the best practical means. This plant (together with the sulphate of ammonia plant) works in connection with the Semet-Solvay ovens of the Wigan Coal and Iron Company. The plant is simple, and with a reasonable amount of attention, gives very good results. By means of this process the sulphur is recovered in the solid form. The essential parts of a Claus plant are shown diagrammatically in Fig. 90.

The waste gases pass first through a coke tower A to remove any excess of moisture, thence into the Claus kiln proper, B. This kiln is filled to a depth of about 3 feet with lumps of iron ore, the bottom layers being in good-sized lumps 3 or

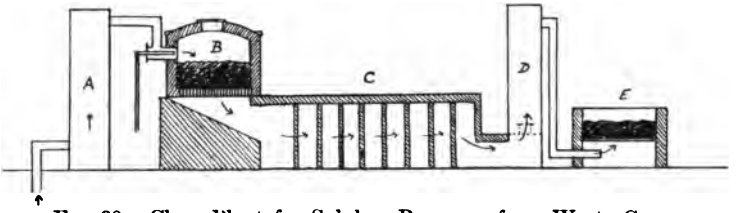


FIG. 90.—Claus Plant for Sulphur Recovery from Waste Gases.

4 inches in diameter, the middle layer rather less, and the top layer of still finer lumps, finished off with a coating of bog iron ore such as is used in the purifiers. The whole is supported on iron grids so as to allow a free passage of the mixed gases through the mass. The principle upon which the reaction in the kiln is based, is that when sulphuretted hydrogen is mixed with a certain volume of air—usually about 10 per cent. is required—and heated to a certain temperature, the following reaction goes on:—

$$H_2S + O = H_2O + S.$$

The initial heat requisite to start the above reaction is supplied by a few shovels full of hot coal or coke put in through the top of the kiln, and, once started, the heat is easily maintained by the catalytic action between the oxide of iron and the mixture of sulphuretted hydrogen and air. The heat is con-

tinuous if the proper relative proportion of these gases is regulated, and this is not at all difficult. If too much air is going in there will be indications of sulphur dioxide which can be recognised by its action on litmus paper. Naturally this means loss of sulphur, as well as the cooling action on the kiln.

$$2H_2S + 3O_2 = 2SO_2 + 2H_2O.$$

Again, if the air supply is deficient, a portion of the sulphuretted hydrogen passes through the oxide without decomposition and may be detected by means of paper soaked in a solution of lead acetate. The slightest trace of sulphuretted hydrogen turns this to a brownish black colour at once, owing to the formation of lead sulphide. The gases may be conveniently tested at two points—one near the kiln, the other near the base of the limestone tower D, through suitable holes left in the brickwork, fitted with wooden stoppers.

When the kiln is working satisfactorily and the reactions going on properly, it is generally found that there are indications of both sulphuretted hydrogen and sulphur dioxide. These gases react in presence of each other thus:—

$$10SO_2 + 10H_2S = 5S_2 + 8H_2O + 2H_2S_5O_6.$$

The sulphur thus set free is in an extremely fine state of division, and the milky effluent from the limestone tower is due to this finely divided sulphur in suspension, and which has most probably been produced by practically the same reaction as above, *i.e.*, by the interaction of sulphuretted hydrogen with either gaseous sulphur dioxide or a solution of the sulphite.

The reaction is also interesting because it most probably represents that which takes place in nature on a large scale, resulting in the deposits of native sulphur usually found in volcanic districts. Both sulphuretted hydrogen and sulphur dioxide are present in volcanic gases. After reaction and deposition of sulphur the remaining gases pass on and upwards through a tower D, which is loosely packed with lumps of limestone, over which a trickle of water is fed from a spray at the top of the tower. This is to neutralise any acidity due to any sulphur dioxide which may remain in the gas. From the tower D the gases are led through purifiers E to remove remaining traces of sulphuretted hydrogen and cyanides.

The sulphur deposited in the first chamber next to the kiln is practically all first quality, and runs down in the molten condition. It may be either run off in this state or allowed to accumulate until a mass 5 or 6 feet deep and weighing 18 to 20 tons is obtained. The sulphur in the later depositing chambers is often found in beautifully crystalline forms, hanging from the roof like stalactites, also in a very fine condition and usually containing a fair amount of moisture. It is quite possible to obtain a product containing 90 to 95 per cent. sulphur, the remainder being moisture, with a trace of ammonium carbonate and sulphate, probably produced by oxidation of the sulphite. It is used by acid makers in the manufacture of brimstone vitriol. In addition to those reactions resulting in the deposition of sulphur there are others going on in the kiln.

The formation of pentathionic acid by the interaction of sulphur dioxide and sulphuretted hydrogen leads to the production of pentathionates, whilst thiosulphates are also formed owing to the action of free sulphur on the sulphites.

Mr R. Forbes Carpenter and Mr Linder have made most thorough and valuable research with regard to the reactions taking place in the Claus kiln. The results of their investigations are given in detail in papers read before the Society of Chemical Industry in March 1903 and June 1904, and they are recorded in minute detail in the *Journal* of that Society.

They proved that hydrocyanic acid reacts with steam, producing ammonia, thus:—

$$HCN + H_2O = NH_3 + CO,$$

and that this conversion took place when a mixture of hydrocyanic acid and carbonic acid was passed through highly heated contact material, and further that the percentage was increased when sulphuretted hydrogen was present. This accounts for the presence of ammonium salts in the recovered sulphur, over and above that which is due to ammonia having escaped the saturators. The Claus plant, therefore, not only is a means of sulphur recovery, but also of purifying the exit gases to some extent from the excessively dangerous hydrocyanic acid. Some, however, remains, and the characteristic smell of cyanide is very noticeable when the kiln is cleared out. When necessary to close down the plant for cleaning it is most necessary to allow

ample time for the gases in the chambers to diffuse out by opening the brickwork about two days before the workmen are allowed to enter; even then every precaution must be taken, as the gaseous mixture hangs about the walls very tenaciously, and is most deadly. A good quality of recovered sulphur should have an approximate analysis as follows:—

Moisture - - - - - -	3 to 5	per cent.
Ammonia, calculated as carbonate	3 to 4	„
Mineral matter, non-volatile -	1	„
Sulphur - - - - - -	90 to 93	„

The second-grade quality contains more moisture and ammonium salt. The ammonia may be determined indirectly by estimating the loss on washing the sulphur with water, or more accurately by distillation with sodium hydrate into normal sulphuric acid. The sulphur may be taken by difference after careful estimation of all impurities. It may also be extracted by carbon disulphide, in which case any amorphous sulphur will be unacted upon. The mineral matter, other than sulphur, is that which remains after burning away the sulphur as completely as possible.

CHAPTER XV.

VALUATION OF AMMONIUM SULPHATE—MANUFACTURE OF CONCENTRATED AMMONIACAL LIQUOR.

The Testing and Valuation of Ammonium Sulphate.—This bye-product, if reasonably pure, is white in colour. In some instances, owing to various defects in the methods of its recovery, the salt is blue, and more rarely brown. In the majority of cases, especially with blue salt, the value is much depreciated on account of colour, but in a few instances, generally with Chinese traders, a brown to black salt is insisted upon, and consequently to meet this requirement a white salt is mixed off with some dark colouring matter to give the desired effect. This, of course, lowers the percentage of ammonia.

Sulphate of ammonia, when absolutely pure and dry, contains 25·75 per cent. of ammonia (NH_3). The bye-product from coke manufacture, if well made, ought to contain 24·5 to 25 per cent. The salt as usually produced contains also a certain amount of moisture, which varies from about ·5 to 2·0 per cent. In addition to this, there is always more or less "free acid" in the salt. This ought not to exceed ·5 or ·6 per cent. If the amount is much more than this it is most likely to cause trouble when the material is loaded up in sacks for transport, owing to the action of the acid on the sackcloth, which eventually causes the bottom of the sack to give way entirely. This is the more likely if at the same time the salt is abnormally wet. The terms of contract for a supply of this material usually stipulate that there shall be a minimum of 24 per cent. of ammonia, and that the salt shall be free from cyanide, by which is meant the ferrocyanide which causes the blue colour.

The estimation of ammonia in sulphate may be made in the same apparatus as that in which the liquor is tested, and the

principle of the test is the same, namely, that the sulphate, of which a weighed portion is taken, is decomposed by the addition of sodium hydrate, its ammonia liberated in the free condition as NH_3, which is passed into a measured quantity of a normal or standard solution of sulphuric acid. From the amount of acid neutralised a measure of the ammonia is obtained, each cubic centimetre of normal sulphuric acid neutralised representing ·017 gram of ammonia (NH_3).

An example of an actual test is as follows:—5 grams of the sulphate were dissolved in water and diluted to 500 cubic centimetres in a flask. After thoroughly mixing, 25 c.c. were taken out by means of a pipette, and transferred to the distillation flask. This portion therefore contained ·25 gram of the salt. A moderate excess of sodium hydrate being added, the distillation was gradually carried on for about three-quarters of an hour into the bulbed tube containing 10 c.c. normal sulphuric acid. When all the ammonia was over, the U tube was detached, and its contents rinsed into a porcelain basin and titrated with a normal solution of soda, 6·35 c.c. being required to neutralise the remaining acid. Hence the acid neutralised by the ammonia liberated from ·25 gram of the salt = 10·0 − 6·35 = 3·65 c.c., and 3·65 × ·017 = ·062 gram NH_3.

That is, ·25 gram contains ·062 c c.

1·00 „ ·248, or 100 contain 24·8 c.c.

That is, 24·8 per cent. ammonia in the salt.

The amount of moisture in sulphate may be determined by weighing off 5 or 10 grams, and drying in a water bath at a temperature of 100° Cent. for about two hours until the weight becomes constant. The loss represents moisture.

The free acid present is determined by dissolving 5 grams of the salt in distilled water, adding a few drops of methyl orange solution and titrating with standard soda. If great accuracy is necessary, it is advisable to use decinormal soda, that is, one-tenth the strength of the normal, or ·004 gram hydrate per 1 c.c.

The Formation of Blue Salt.—As previously mentioned, the occurrence of blue salt is not uncommon under certain working conditions, and much time and thought have been spent in

endeavouring to prevent its formation, more particularly because the colour affects the marketable value.

The cause of the blue colour was not very difficult to diagnose. It evidently arose from the presence of a ferrous salt and a ferrocyanide in the saturator liquor. The presence of the iron salt could be accounted for in several ways. For instance, it might be originally present in the sulphuric acid used to absorb the ammonia, or the acid might have come in contact with iron or steel in some part of the plant, but the presence of ferrocyanide was not so easy to explain. For those who are interested sufficiently in the matter, reference may be made to the Report of the chief Alkali Inspector to the Local Government Board, 1905, in which Messrs Linder, Young, and Sutton record their research and observations. It is the experience of the authors that local alkalinity in the saturator, due to a defective blow-pipe, for instance, may be one cause, and that certainly the bath ought always to be kept on the acid side, at the same time the temperature must be kept up by steam. Under these conditions blue salt is practically impossible.

The Formation of Yellow Salt.—This is undoubtedly due to the presence of arsenic sulphide. It is not produced so commonly as the blue salt. The arsenic is derived from the sulphuric acid used, and therefore it is necessary to examine the acid for arsenic.

The sulphuric acid used in the production of sulphate of ammonia should be practically free from impurities, especially iron, arsenic, and total solid residue, principally lead sulphate. The amount of total solids can be readily ascertained by evaporating a measured quantity in a weighed platinum dish —this should be done in a good draught cupboard—and finally heating up to dull redness in the gas muffle, or over a good Bunsen flame. If the amount of solid residue is excessive, the quality of the sulphate suffers.

The iron may also be conveniently estimated in this residue by digesting it with concentrated hydrochloric acid until all the iron oxide is dissolved, then either determining volumetrically by means of $\frac{N}{10}$ potassium dichromate or permanganate, or by

filtering off any insoluble matter, separating any lead by means of sulphuretted hydrogen, then precipitating the iron in the filtrate, after oxidation with a little nitric acid, by means of ammonium hydrate, boiling, filtering, washing, and finally after ignition weighing as ferric oxide. The volumetric method is much simpler and quite accurate. If the amount of iron is excessive it increases the likelihood of the formation of blue salt (see page 120). The arsenic may be readily detected by Marsh's test, which depends upon the fact that when nascent hydrogen is liberated in any solution—as when zinc is added to dilute sulphuric acid—any arsenic which may be present comes off as arseniuretted hydrogen. If the gas thus generated in a flask be conducted through a glass tube drawn out to a jet, and lighted, after all the air has been expelled, the arsenic shows at once on any cold surface held in the flame. A porcelain dish or crucible lid is most suitable, and a black, lustrous, metallic-looking deposit is formed if the slightest trace of arsenic be present. The zinc used must be specially free from arsenic. The presence of arsenic in any quantity leads to the production of yellow salt, due to the formation of sulphide of arsenic.

An average amount of the above impurities in the sulphuric acid used is as follows:—

Total solids - - - - -	·05 to ·07 per cent.
Ferric oxide - - - - -	·015 to ·035 „
Arsenic - - - - -	*nil* to traces.

The specific gravity usually runs about 1·74, and is generally used lower in winter than summer.

Manufacture of Concentrated Liquor.—This method of working up the ammoniacal liquor on coke works is now being extensively adopted, and in many respects possesses advantages over the sulphate method. No acid is required, and as no waste gases are given off, the expense of purification is dispensed with. The plant as a rule requires very little attention, takes up comparatively little room, and less labour is required in the working of it. The principle is very simple, consisting of (*a*) distillation of the coke works liquor, as in sulphate manufacture, and (*b*) condensation of the vapours produced, and absorption of

ammonia. A very efficient type of plant, manufactured by Messrs Brunner, Mond, & Co., is shown in Fig. 91. It consists of a still K, and a series of absorbing vessels A, B, C, D, E, and a

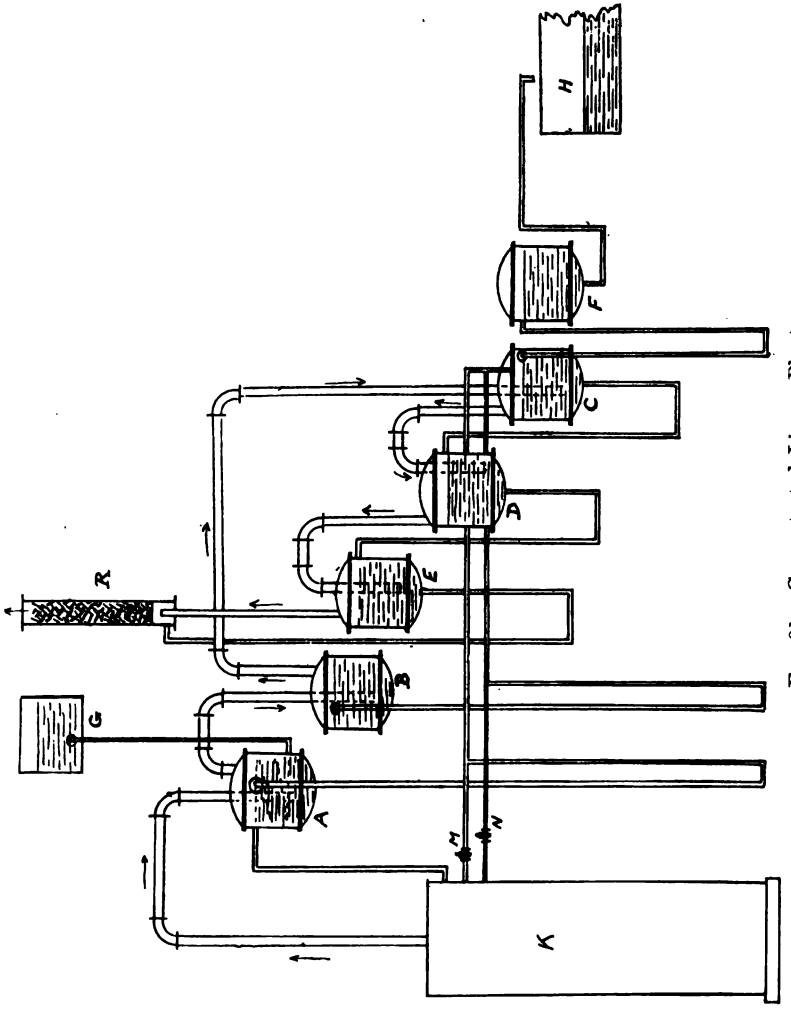


FIG. 91.—Concentrated Liquor Plant.

cooling vessel F. The vapours from the still pass through the absorbing vessels, each of which contains about 2 feet of liquid. Thus the total depth of liquid through which the vapours are forced is about 10 feet, as opposed to about 2 feet of acid in

sulphate making. The pressure on the top of the still is thus higher in the case of concentrated liquor, and provision for this must be made in designing the still. In the plant described the gases from the still pass into the first absorbing vessel A. Here they are cooled by cooling coils, through which cold water or (as shown in the drawing) cold weak liquor is run. Thus the first vessel may be used practically as a superheater. As the bulk of the condensation takes place in this vessel, a great proportion of steam is condensed, and the liquor from this vessel is usually run back into the still through the cock M. The temperature in this vessel is too high for complete absorption of ammonia, and the gases pass on through the other absorbing vessels B, C, D, E, in the order named. The temperature in these vessels is easily controlled by cooling coils (not shown in sketch), and the whole of the ammonia is absorbed. The gases, if any, from the final absorbing vessel E pass through a coke tower R, down which a small stream of cold water runs. This water takes up the last traces of ammonia or sulphuretted hydrogen, and passes on through D to C, taking up more ammonia in its passage. It will be seen that the liquor from B also flows to C. Thus the liquor in C is the final liquor, but to avoid loss by volatilisation, the liquor is cooled in vessel F before passing to the store tank H.

Besides ammonia, carbon dioxide and sulphuretted hydrogen and hydrocyanic acid are absorbed in the various vessels, and the final concentrated liquor is really a mixture of ammonium carbonate, ammonium sulphide, ammonium hydrate, ammonium cyanide, &c.

The total ammonia in the liquor by the above plant may be easily brought up to 18 per cent., but it is not advisable to go beyond this point, as the tendency to crystallise increases rapidly, and there is danger of stoppages in the pipes, chiefly through ammonium carbonate. In satisfactory working the pressures on each vessel should show a gradual decrease to atmospheric pressure, and the temperatures on the vessels should also decrease uniformly. The following figures show an average working:—

Pressures :—

Top of still.	A.	B.	C.	D.	E.	
12.	9½ to 10	7½ to 8	5 to 6	3 to 3½	0 to 1	in. of mercury.

Temperatures :—

Top of still.	A.	B.	C.	D.	E.
100° Cent.	80 to 85°	70 to 75°	50 to 55°	40° Cent.	25° Cent.

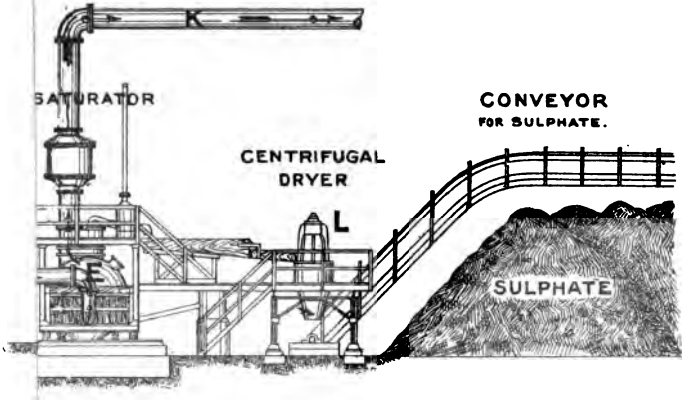
Per cent. of ammonia :—

A.	B.	C.	D.	E.
10 p. cent.	15 p. cent.	17 p. cent.	13 p. cent.	4 p. cent. NH_3.

CHAPTER XVI.

Recovery of Ammonia direct from Coke-Oven Gas.

Recovery of Ammonia direct from Coke-Oven Gas.—The manufacture of sulphate of ammonia or concentrated ammoniacal liquor, as described in the last chapter, requires rather costly plant in order to thoroughly condense the water vapours in the gas, and to completely remove the ammonia by various forms of scrubbers. Attempts have been made to recover the ammonia by passing the gas directly through a bath of sulphuric acid. These attempts, in the first instance, proved unsatisfactory owing to difficulties with the saturator, and also to the incomplete removal of the tar. However, the tar may now be completely removed by the modern types of tar extractors, and by careful control of the temperatures of the gas, the difficulties connected with a hot acid bath may be dispensed with. The process for direct recovery of ammonia from coke-oven gas as here described, has been recently patented by the Koppers Coke Oven and Bye-product Company. In this process the temperature of the gas is so adjusted by a superheater as to prevent undue condensation of the vapours, which pass forward directly into an acid bath at a comparatively low temperature. The plant, as shown in Fig. 92, consists of a cooler A and water coolers B, which reduce the temperature of the gas to about 25° Cent. At this temperature the whole of the tar can be removed by the tar extractor D, the gas being circulated by the exhauster C. The gas, freed from tar, passes through the cooler A, where it exercises a cooling effect on the hot gases, becoming in itself heated to a temperature of 40° to 50° Cent. The affinity between ammonia and sulphuric acid at this temperature is greater than in ordinary sulphate manufacture (105° Cent.), and a neutral salt is easily produced. The liquor collected from the coolers and water condensers



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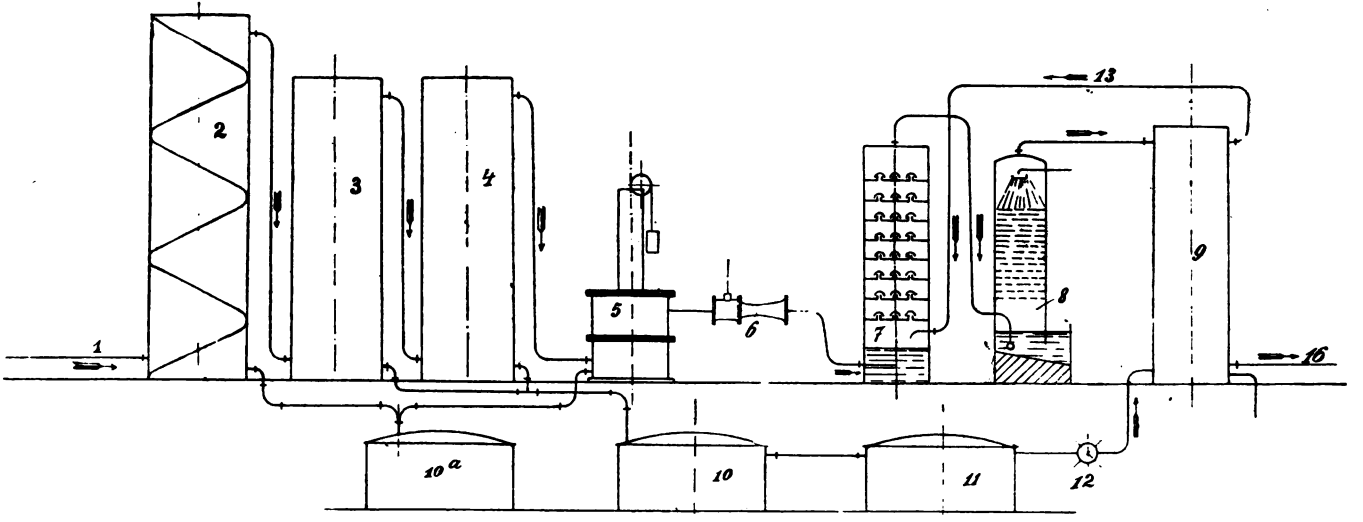

FIG. 93.—PLANT FOR THE DIRECT SATURATION OF GASES. (Coal Distillation Co.).

(after decantation from tar) is distilled in the usual way, the ammonia vapours passing into the gas main F, and thus into the saturator. The sulphate is ejected from the saturator and dried in the usual manner. The sulphate is free from tarry matter, of good grey colour, and of good quality, as shown by the analysis:—

Moisture - - - -	1·86 per cent.
Free acid - - - -	·37 „
Ammonia - - - -	25·05 „

There are many advantages attached to this system. In the first place, scrubbers are dispensed with, and the amount of cooling water is greatly reduced. The amount of liquor requiring steam for distillation is less than half the usual amount, and the quantity of waste effluent is correspondingly reduced. The purification of the waste gases from the usual sulphate plant is avoided. It may be stated that, owing to the low temperature in the saturator, the valuable hydrocarbons of the gas are not destroyed.

The type of plant recently patented by the Coal Distillation Company of Middlesborough for direct saturation of gases is shown diagrammatically in Fig. 93. The gas passes through the condenser 2, giving up the greater part of its tar in a condition free from water. The gas then passes through water condensers 3 and 4 and the tar extractor 5. The tar from 2 and 5 is collected in the tank 10*a*, whilst tar and ammoniacal liquor from the water condensers are collected in 10 from which the ammoniacal liquor overflows to tank 11. The jet exhauster 6 draws the gas through the above portion of the plant, and forces it through the still 7 and saturator 8. The liquor from 11 is pumped through the condenser and superheater 9 into the still 7, where the steam used in the jet exhauster serves a useful purpose in distilling it. The hot gases containing ammonia vapour, pass through the saturator 8, the last traces of ammonia being taken out in the tower extension which is lead-lined and filled with acid-proof material, down which sulphuric acid flows. The ammonium sulphate is collected in the usual manner. This plant also dispenses with the use of scrubbers, whilst the amount of steam used is considerably lessened by making use of the steam introduced in the jet exhauster.

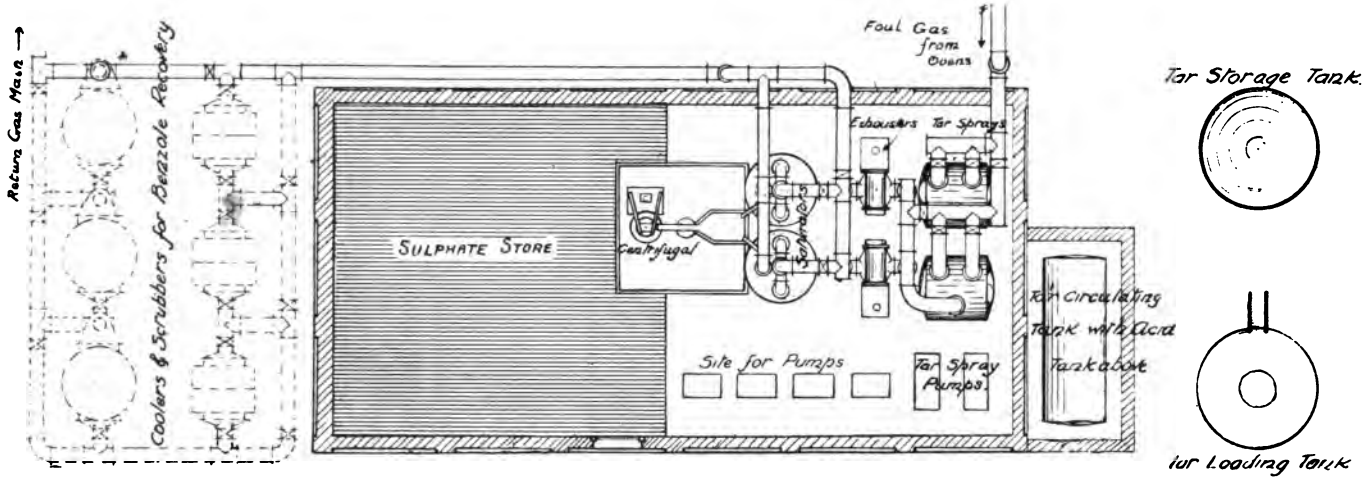

FIG. 94.—THE OTTO-HILGENSTOCK SYSTEM OF SULPHATE MANUFACTURE.

Complete recovery of ammonia is ensured by use of the acid scrubber.

The Otto-Hilgenstock Coke Oven Company have also recently introduced a patent process of sulphate manufacture in which the gas is passed direct through a bath of sulphuric acid. This system shows a remarkable improvement in the direction of economy and simplicity. As shown in Fig. 94, the hot gas from the ovens is drawn by means of exhausters through a set of two tar sprays, which remove the whole of the tar from the gas before any condensation of ammoniacal liquor takes place. The gases are then passed directly into the saturators, no cooling whatever being required. The complete removal of tar ensures a good grey salt. A very important point in this process is the fact that no cooling water is required. Also no ammoniacal liquor is produced, as the gases pass into the saturators at a temperature high enough to prevent any condensation. Consequently, no lime, or steam, is required for distillation purposes. At the same time no waste liquors (requiring settling tanks) are produced, and the annoyance and trouble in dealing with these are entirely removed. In addition to these, water condensers, scrubbers, &c., are no longer required, and the total space required is remarkably small in proportion to the rest of the coking plant. The sulphate from the saturators is dealt with as described in the last chapter.

CHAPTER XVII.

Use of Surplus Gas from Coke Ovens—Determination of High Temperatures.

Surplus Gas from Coke Ovens.—The use of the gas from coke ovens is now a very important question. All modern retort ovens give off more gas than is required for heating the oven. In the "waste heat" type of oven the amount in excess is usually 20 to 30 per cent. of the total production, whilst in ovens using regenerators this amount is increased to as much as 50 per cent. With slack containing about 30 per cent. volatile matter, the total gas evolved may be taken as 9,000 to 10,000 cubic feet per ton of coal. Thus in ovens using regenerators there is a surplus of 4,500 cubic feet per ton of coal, which may be used as follows:—

(*a*) Lighting purposes.
(*b*) Steam raising, by burning the gas under boilers.
(*c*) Power generation, by combustion in gas engines.

Dealing with the first method, the candle power of coke oven gas is most important, having regard to the restrictions imposed by the authorities in this respect. The lighting power prescribed by Act of Parliament is usually about 14 candles. The candle power of coke oven gas from an individual oven gradually decreases from the time of charging to the time of discharging an oven. Taking the earlier portion of the coking period, the lighting power of coke oven gas averages 14 to 16 candles, whilst the later portion produces a gas of an average candle power of 7 to 9 candles. Accordingly, several coke oven plants in America have adopted a system in which two gas-collecting mains are used—one for the "rich" gas given off during the earlier portion of the coking period, and the other for the "heating" gas given off during the latter portion. The

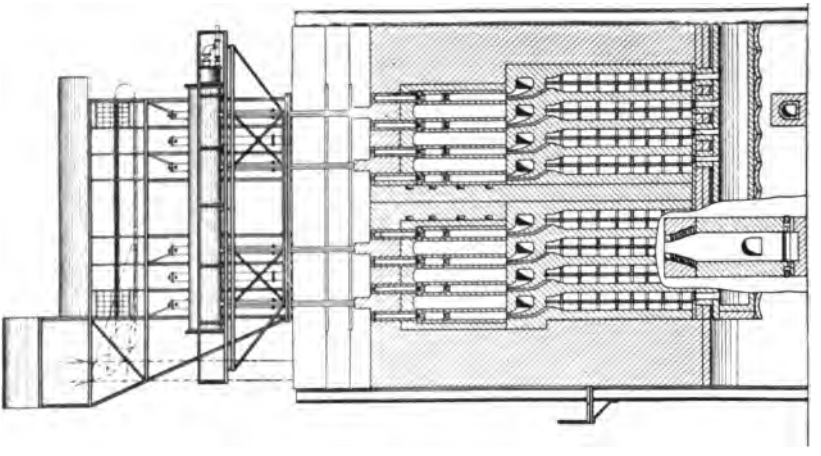

FIG. 96.

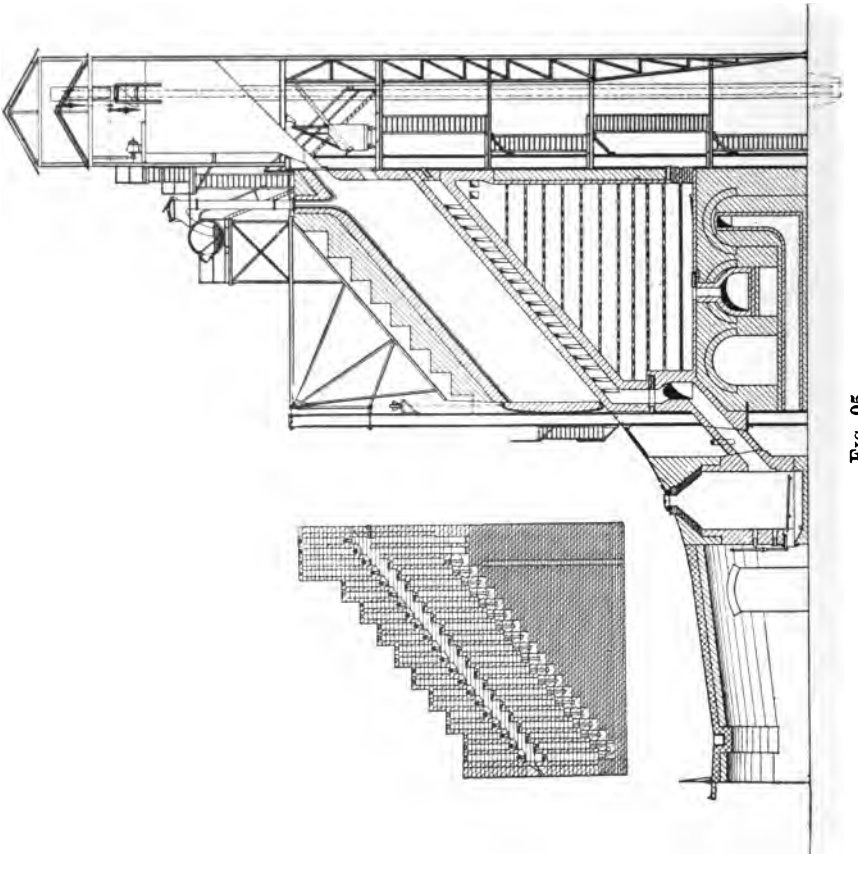

FIG. 95. THE KOPPERS INCLINED OVEN.

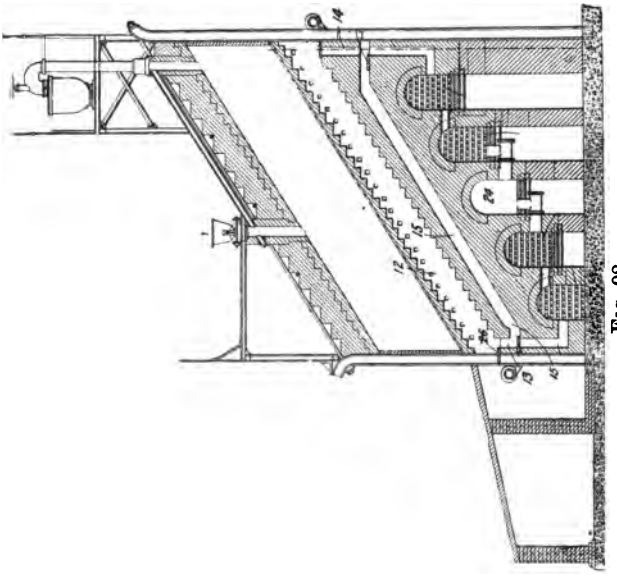

FIG. 98.

THE COLLIN INCLINED OVEN.

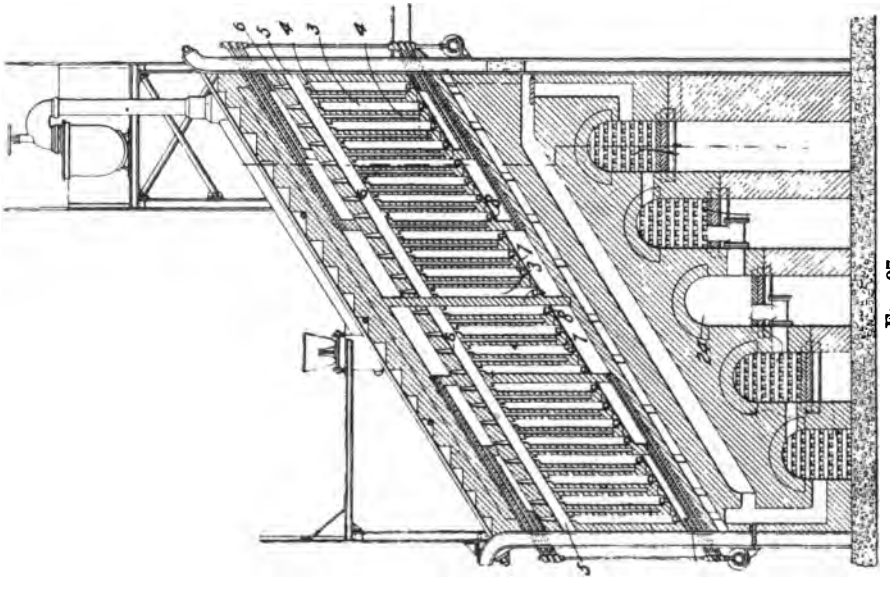

FIG. 97.

"rich" gas is led through special purifying plant, and is delivered to the neighbouring towns. The "heating" gas is used in the oven flues for carrying on the coking process. The calorific power of this heating gas averages (with Lancashire coals) 500 B.T.U. per cubic foot, enough to maintain quite easily the necessary flue temperature. Thus, with suitable precautions, there is a prospect of a combination of coke works and gas works practice in the near future, whereby good, hard, metallurgical coke will be produced, as well as gas suitable for lighting purposes. In taking off rich surplus gas for this purpose the ovens should be built with all possible care to ensure gas-tightness, and with better arrangements for rendering the doors air-tight. The gases should be led off from the charge as quickly as possible to prevent undue contact with the hot sides and roof of the oven, as this would bring about a splitting up of the illuminants in the gas. The ovens should be completely filled or nearly so, in order to reduce the area of red-hot surface. Coke ovens are now being laid down on the Continent on the above lines, and the Koppers inclined coke oven (Figs. 95, 96) and the Collin oven (Figs. 97, 98) are designed on the lines of the inclined retorts of gas works practice, whilst retaining the particular features of each type of oven as regards the arrangement of flues, &c. The whole question of the use of coke oven gas for lighting purposes has been dealt with very fully by Mr Ernest Bury, M.Sc., F.C.S., in a paper read before the Institute of Gas Engineers in June 1907, the subject-matter of the above paper being in close accordance with the authors' experience.

The second method has up to recently been largely adopted with ovens of the waste heat type, the surplus gas being fed into the boiler tubes through suitable Bunsen burners, the heat from this surplus gas thus augmenting the waste heat from the ovens. It has been found, however, that by using the gas in gas engines the power derived is between twice and three times the power derived by burning the gas under boilers. Consequently the surplus gas has received more attention of late years, and the gas engine has been adopted by several firms with a fair amount of success. The difficulties at first met with on the introduction of the gas engine are being gradually overcome. The tar and naphthalene which interfered with the

FIG. 99.—KOPPERS OVENS AT BARGOED COLLIERY, SHOWING GAS-HOLDER.

working of the valves are now removed by more modern extractors and washers. The sulphur compounds, which gave rise to acid products of combustion, and consequently serious corrosion, are completely removed by passing the gas through purifiers, which also remove any particles of dust. The high proportion of hydrogen in coke oven gas tends to cause pre-ignition, but this has been largely overcome by careful attention to the regulation of the degree of compression of the gas. Gas engines, to ensure the maximum economy, should be worked at as steady a load as possible, and the quality of the gas should be kept as uniform as possible. It is therefore advisable to use a gas-holder, which also stores the gas evolved in the night-time to be used in the day-time, when the power required is usually greater. The following is a brief description of the plant erected by the Powell Duffryn Steam Coal Company at Bargoed Colliery, South Wales, in connection with Koppers coke ovens.*

The oven plant consists of two batteries of fifty ovens each of the Koppers type. The gas given off amounts to 10,000 cubic feet per ton, with a calorific power of 460 B.T.U. per cubic foot, and of the following analysis:—

Hydrogen	63·42 per cent.
Methane	23·14 „
Carbon monoxide	5·21 „
Carbon dioxide	2·01 „
Ethylene	·80 „
Oxygen	·42 „
Nitrogen	5·00 „

The illuminating power of the gas is low if used in an ordinary bat's-wing burner, but when applied to an incandescent gas mantle the light is very brilliant. The first gas engine set laid down was of the Nürnberg type of 1,200 B.H.P. This has worked quite satisfactorily in parallel with steam sets, and the Colliery Company have since put down a larger set of the same type to develop 2,400 B.H.P. To provide for fluctuations in the production of gas, a gas-holder of 300,000 cubic feet has been erected. The supply of electric power extends over all

* From a paper read before the South Wales Institute of Engineers by Mr E. M. Hann, M.I.C.E.

the Company's collieries in the Rhymney Valley, and includes altogether 170 motors, eleven of which are haulage motors of 50 to 250 H.P. The Company have also installed a gas engine of the Cockerill type, of 650 H.P., to drive a ventilating fan.

The gas for the above plant is purified by passing first through the usual bye-product recovery plant and secondly through oxide purifiers, of which there are two groups. Each group consists of four boxes, each box 30 feet square by 6 feet high, containing layers of hydrated oxide of iron on wooden grids, as in the ordinary method of gas works purification. The oxide is revivified and re-used (page 114) until it contains about 60 per cent. sulphur. This mixture is then burnt, and the sulphur converted into sulphuric acid. The waste gases from the sulphate plant are also burnt in the same furnace, and the combined yield of sulphur gives sufficient sulphuric acid to satisfy the requirements of the sulphate house. The tests on the 1,200 H.P. engine, working under full load of 820 kilowatts with gas of 458 B.T.U., showed a consumption of 25·533 cubic feet per hour, or 21·3 cubic feet per B.H.P. hour.

Gas Analysis.—For the analysis of coke oven and other gases the authors have found the apparatus designed by Mr Stead to be very suitable. It is illustrated in Fig. 100.

The reservoir on the left of the diagram contains mercury, and this is connected with the eudiometer and levelling tube by means of the U-shaped joint. The eudiometer or measuring tube is graduated in millimetres, and has platinum wires fused through the upper portion for the explosion of gaseous mixtures. The tube of the eudiometer is drawn out at the top to a bore of about 3 millimetres, and is connected to a three-armed capillary tube, to which three absorption vessels may be attached (only one is shown in the figure). The capillary tube is also extended to the right, and through this the sample of gas is taken into the eudiometer. It will be seen that by raising the mercury reservoir, by means of a cord holding a counterweight and passing over pulleys, the mercury will flow into the eudiometer, and, the tap being opened, any gas or air in the tube will be completely expelled. The gas sample, or bottle containing the sample of gas, may now be attached to the end of the capillary tube, and by lowering the mercury reservoir any desired volume

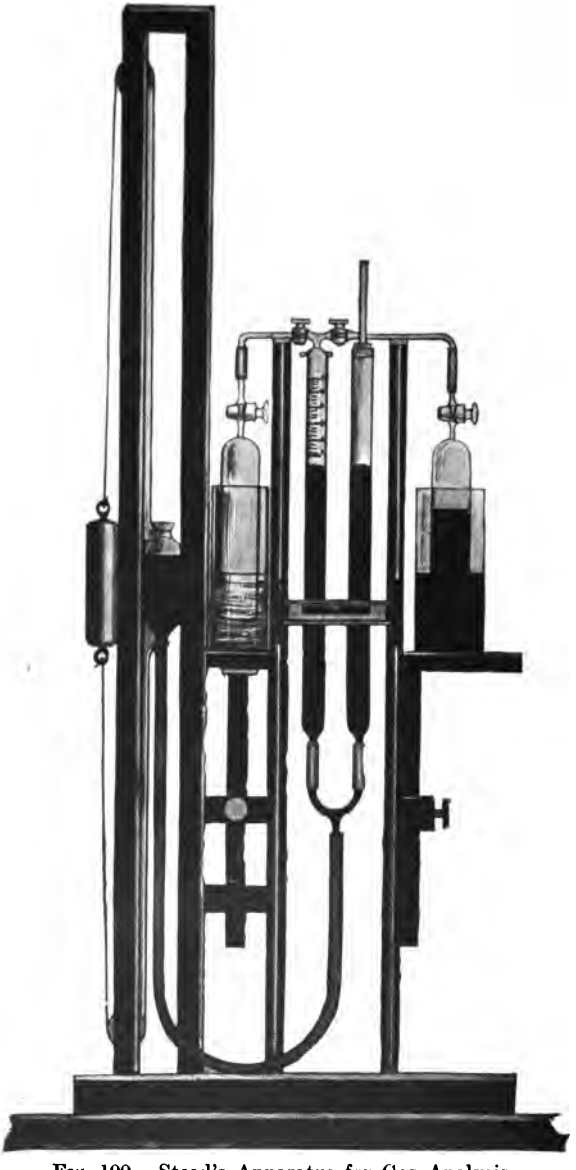

FIG. 100.—Stead's Apparatus for Gas Analysis.

may be drawn into the eudiometer for analysis, and the tap shut off. By means of the levelling tube the pressure is kept

constant, since it is open to the atmosphere; and also, by means of a water jacket round the eudiometer (not shown in the figure), the temperature is constant, thus avoiding corrections otherwise necessary. The gas, after measurement, is passed into the various tubes connected to the three-armed piece containing various absorbents. It will be readily seen that the gases may be transferred from one absorbing vessel back to the eudiometer for measurement, and then into other absorbents as desired, by manipulating the mercury reservoir. In an analysis of oven gas the following would be the procedure:—

Take a measured quantity of gas, say about 200 cubic centimetres, into the eudiometer, measuring carefully with the mercury at the same level in the eudiometer and levelling tube; then pass over into one of the absorbing vessels containing a solution of potassium hydrate, leaving it several minutes in contact; then pass back into the eudiometer for measurement. The diminution in volume is due to the absorption of carbon dioxide. The gas is then passed into fuming sulphuric acid or bromine solution, which absorb olefines and benzines, but before final measurement any vapours of sulphur trioxide or bromine must be got rid of by passing over into the potash vessel. The next absorbent used is an alkaline solution of pyrogallic acid (pyrogallate of soda), which takes up oxygen, and finally cuprous chloride is used to absorb the carbonic oxide. An extra absorbing vessel can readily be attached to one of the three arms, or detached to be replaced by any special absorbent. The remaining gases now consist of marsh gas or methane, hydrogen, and nitrogen. To determine the two former, they are mixed with a certain volume of air and an electric spark passed across the platinum wires in the eudiometer. By this means the methane becomes converted into carbon dioxide and water and the hydrogen becomes water. There is a contraction in volume after explosion, and this is carefully measured, and then the mixture is passed into the potassium hydrate to absorb the carbon dioxide, which is measured by the diminution in volume after several minutes' contact. It is advisable to take about 20 to 25 c.c. of gas for explosion after removal of carbonic oxide, &c., by absorbents, and dilute with about 200 c.c. of air free from carbon dioxide. If oxygen be used the explosion would be violent enough to burst the eudiometer. The methane, when

exploded with air, forms its own volume of carbon dioxide, hence the last absorption represents the volume of methane present. But in exploding, methane requires twice its volume of oxygen. Therefore by taking twice the volume of carbon

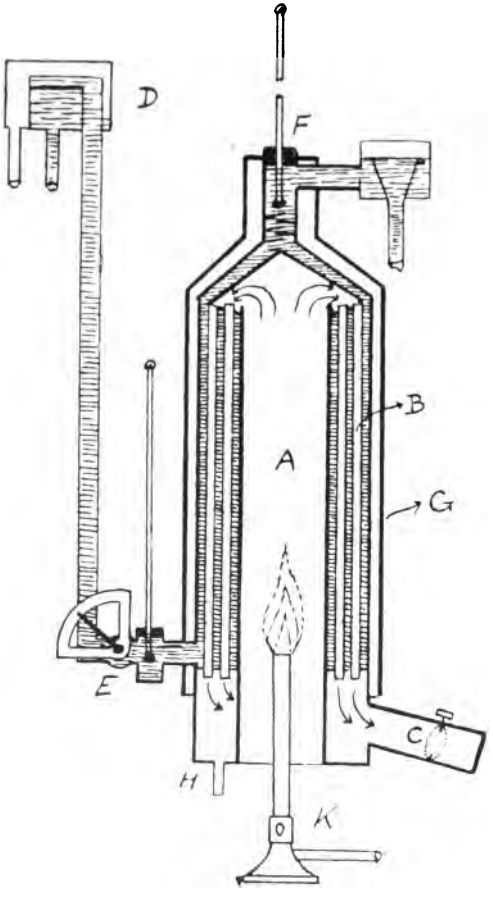

FIG. 101.—Junker's Gas Calorimeter, in Section.

dioxide formed, from the total contraction, the difference is the contraction due to the explosion of the hydrogen, and two-thirds of this contraction is due to hydrogen.

The nitrogen is always estimated by difference. An average

analysis of oven gas from a Semet-Solvay plant is given on page 152.

The chemical composition of the gas produced by a modern coking plant has been already discussed. In addition to this it is important to know its calorific value or heating power. This may either be calculated from its chemical constituents or, which is far preferable, subjected to an actual test in some form of calorimeter. Of these there are several kinds, but broadly speaking they depend upon the same principles. The particular instrument with which the authors are acquainted is known as Junker's gas calorimeter, and is illustrated in Figs. 101, 102. The principle of the apparatus is, that the heat generated by the flame of the gas under examination, and which is burnt in a Bunsen burner, is transmitted to a current of water flowing at a constant rate, and measurements are taken of the quantity of gas burned by passing it through a small meter. The quantity of water passed through the apparatus is also measured, as well as the difference in temperature between the water coming in and that flowing out. The gas is burnt in an annular vessel of copper, the annular space being occupied by a number of copper tubes which connect the top with the bottom of the chamber. The heated products of combustion pass along these tubes in one direction, and a current of water passes outside them in an opposite direction. By these means the whole of the heat of the gases is taken up by the water, after which they pass out

FIG. 102.—Junker's Gas Calorimeter.

into the atmosphere. Since the gas contains marsh gas and hydrogen, steam is produced by their combustion: thus steam becomes condensed, runs down to the lower part of the apparatus, and is there run off into a graduated vessel and its volume noted. The whole body of the instrument is enclosed in an air jacket consisting of a plated copper cylinder, to prevent radiation of heat. An example is appended giving the figures of an actual test:—

Volume of gas by meter - - -	6,500 c.c.
Temperature of gas - - -	22° Cent.
Normal pressure - - - -	760 mm.
Pressure of gas examined - -	20 mm.
Water collected from 6·5 litres -	2 c.c.
Water used for cooling - - -	2.770 c.c.
Temperature of inlet water - -	19·45° Cent.
„ „ outlet „ - -	29·20° „
Increase -	9·75° Cent.

Then the corrected volume of gas will be—

$$6{,}500 \times \frac{780}{760} \times \frac{273+0}{273+22} = 6{,}170 \text{ c.c.}$$

The heat produced is—

$$\frac{2{,}770 \times 9{\cdot}75}{6{,}170} = 4{,}377 \text{ calories per cubic metre,}$$

less latent heat due to water formed during combustion—

$$60 \times 3 = 180$$

$$4{,}377 - 180 = 4{,}197 \text{ calories per cubic metre.}$$

$$4{,}197 \times {\cdot}11236 = 471{\cdot}6 \text{ British thermal units per cubic foot.}$$

The Determination of High Temperatures.—It is often desirable, in fact necessary, to ascertain the temperature in the flues, &c. This may be done in several ways. One very ready and convenient method is by means of Seger's cones or pyroscopes. These are made of material consisting of silicates of varying composition moulded into the form of tetrahedra or triangular pyramids. By varying the proportions of the ingredients used in making the cones, it is possible to alter the temperature at which they fuse. There are some fifty-eight different mixtures having melting points ranging from 590°

Cent. to 1,850° Cent. By subjecting a few of these to the temperature the degree of which it is desired to estimate, and noting the effect upon them, it is easy to determine, quite accurately for all practical purposes, what the temperature is. The Watkin's heat recorders are based on somewhat similar principles. They consist of blocks of refractory material with circular recesses sunk in the top face. In these recesses are placed small pellets of fusible materials of definite composition and melting point, the latter having been determined by means of a standard electrical pyrometer. The illustration (Fig. 103) shows a small recorder with five recesses; the graduated effects of the heat to which it has been subjected are very noticeable. By means of these recorders degrees of temperature from 590° Cent. to 1,870° Cent. may be determined.

FIG. 103.
The Watkin's Heat Recorders.

Electrical Pyrometers.—Another means of measuring temperatures is found in electrical pyrometers. One type of these depends upon the use of a thermo-element constructed of two rods of different materials which are fused together at their extreme ends, the other ends having terminals for wire attachments to a galvanometer. A difference of potential is produced when these rods are heated, causing a current to flow, varying in strength according to difference in the intensity of the heat. The relation of this current to the temperature has been determined, and the scale of the galvanometer accurately divided to read directly in degrees Fahrenheit or Centigrade.

Another type, known as the resistance pyrometer, depends upon the principle that a heated wire offers more resistance to the passage of an electric current than a colder wire of the same dimensions does. The essential part of these instruments consists of a fine wire, usually of platinum, which is wound on a mica frame. As its temperature is increased its resistance becomes greater, and from the amount of resistance offered the temperature is determined. The wire is enclosed in a tube of porcelain, brass or steel, according to circumstances, and is connected by stouter wires to the indicator. Usually

they are constructed to give direct readings of temperature in Centigrade or Fahrenheit degrees.

Thermo-electric Pyrometers are also used. Their action is based upon the fact that when a junction of two dissimilar metals is heated, that an electromotive force is developed proportional to the difference in temperature between the two ends of the "couple." The metals forming the "couple" are usually either platinum and platinum-rhodium, or platinum and platinum-iridium. The electromotive force developed may be measured directly on a galvanometer or a milli-voltmeter, which has been carefully standardised to record correct temperature.

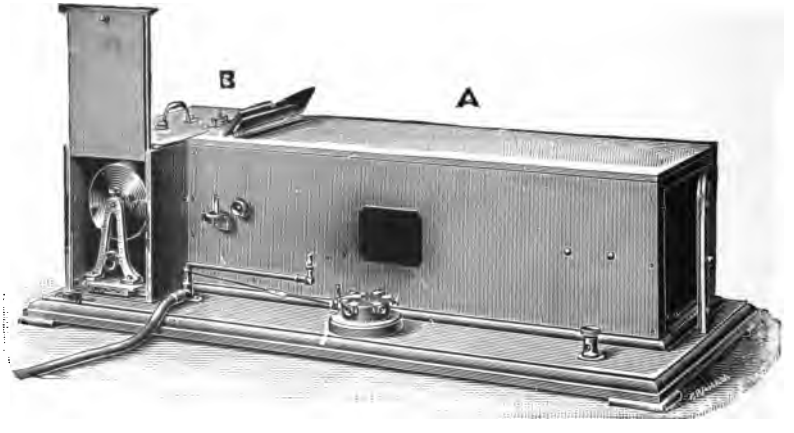

FIG. 104.—The Roberts-Austen Pyrometer.

The Roberts-Austen Pyrometer.—A most ingenious and useful application of this development of an electric current by means of a thermo-electric couple is found in the Roberts-Austen pyrometer. The apparatus is illustrated in Fig. 104, and consists of two light-tight mahogany cases. The larger one A contains a D'Arsonval dead-beat galvanometer, to which is attached a small mirror. The other case B contains a drum which revolves by clockwork, one revolution in every six, twelve, or twenty-four hours as required. Round this drum is fastened a piece of sensitised paper (rapid bromide). When the thermo-couple is heated the current developed passes through the gal-

vanometer, and deflects the mirror in proportion to the intensity of the current. By means of a mirror and a gas jet, shown at the side, a ray of light is thrown on to the galvanometer mirror, and this is reflected back through a slot at the end of A, and acts on the sensitised surface on the drum in B. The deflection of the galvanometer mirror varies with the temperature of the thermo-couple which is placed in the flue or other source of heat the temperature of which is required, and consequently any variation or fluctuation is recorded on the bromide paper, which is developed in the usual way at the end of the twenty-four hours. A scale is also provided in the top of the case A, near B, whereby direct reading of the temperature may be taken at any time, as portion of the light from the galvanometer mirror is reflected upwards on to the ground glass of this scale in the form of a thin band of light. By means of clockwork, this pyrometer is arranged to record automatically, the temperature of three sources of heat during the same period of time, or any one of six sources of heat may be recorded by means of switches.

The apparatus must be kept as free as possible from vibration, and is usually fitted up in a laboratory or office, with connecting wires to the various flues, &c., the intensity of heat in which it is required to measure.

"Black Ends" in Coke.—"Black ends" have always been a source of annoyance in bye-product coke manufacture. It will be seen from the chapters dealing with the construction of coke ovens in general, that in every case the heating of the oven takes place along the sides, leaving the ends of the oven doors exposed to the cooling action of the atmosphere. Experience shows the cooling action to be very considerable, and this results in a layer of uncoked material behind the door, sometimes extending to a depth of several inches, whilst the coke in the interior of the oven is completely burnt off. In ordinary practice this "black end" is raked off, or allowed to burn off to some extent, after opening the door and before discharging the oven. This, whilst it entails a certain loss in the yield, is not always efficient, and the appearance of an otherwise good batch of coke is often completely spoiled by

a relatively small quantity of "black end." Recessed doors have been tried for the purpose of removing the "black end." These lessen the output of the oven to some extent, and are only a partial remedy of the evil. Various forms of flued doors have also been used, some of them quite successfully. In one type (Beach's Patent) the flue in the door is heated by a gas jet at

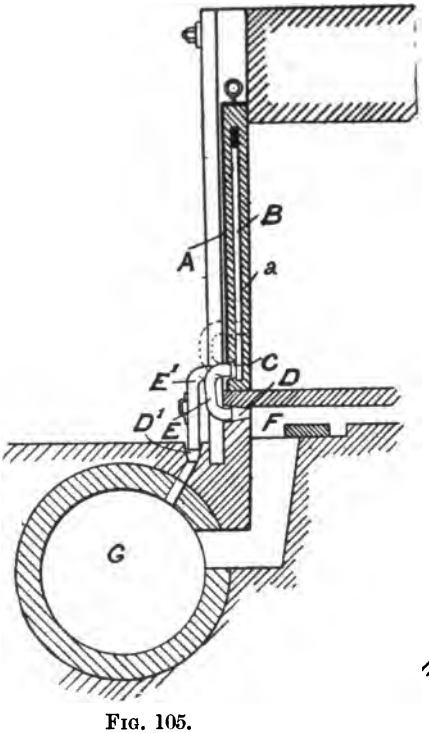

FIG. 105.

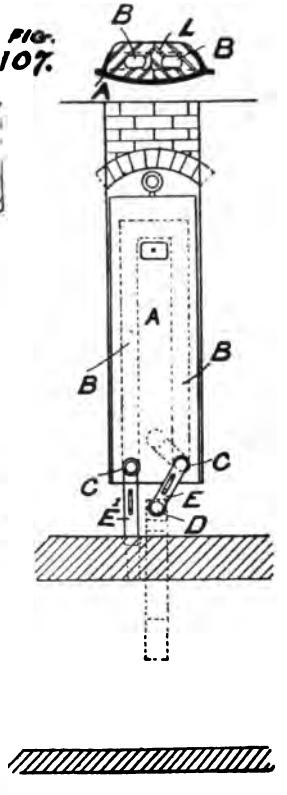

FIG. 106.

the bottom, the waste products of combustion passing into the atmosphere at the top of the door. This, whilst quite efficient, necessitates the use of live gas, and the door now described was designed to avoid this. In the drawing shown (British Patent 17,647, Percy, Shaw, & Entwistle) it will be seen that the waste heat from the oven flues is made use of, being diverted through

the bridge pipes E[1] and E by partially closing the sole flue damper F. Another modification of this patent is simpler in action, requiring less apparatus and less alteration in the oven fabric. This consists in having only one flue of oval section down the middle of the door. The upper end of this flue has a direct connection to the interior of the oven A (Fig. 108), whilst the lower end is connected by the bridge pipe to the sole flue F. In this case only one bridge pipe is required. The bridge pipe is only coupled to the door during the last stages of the carbonising period when practically all the gases have been exhausted from the charge, and only the portion immediately behind the door is uncoked. The heated products of combustion from the oven, assisted by the chimney draught, soon heat the door, and it has been found that the last hour is sufficient to carbonise the ends of the charge completely. Thus the loss of bye-products is reduced to a minimum, and an advantage is gained by diverting the hot gases of the oven to the boilers or regenerators, avoiding unnecessary heating of the hydraulic mains, &c., and a consequent increase in the amount of cooling water used in the condensing plant. The illustration shown represents the latter type of flued door.

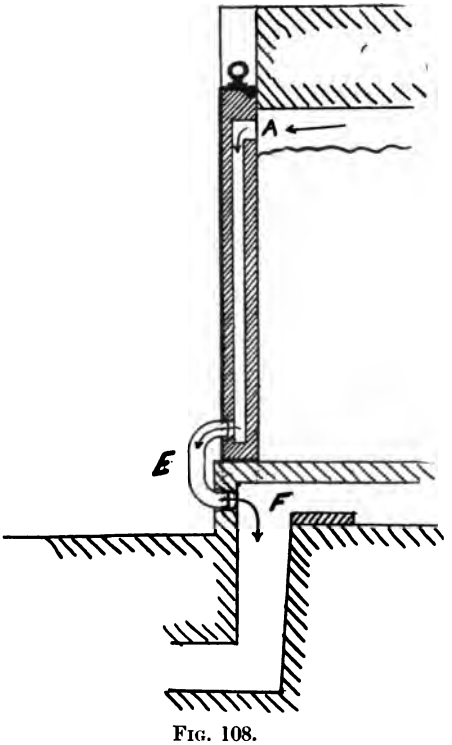

FIG. 108.

FIG. 109.

USEFUL TABLES.

Miscellaneous Specific Gravities.

Platinum	21·5	Boxwood	1·15
Gold	19·3	Asbestos	3·07
Mercury	13·6	Bricks	1·8 to 2·4
Lead	11·4	Portland cement	1·76
Copper	8·9	Slate	2·5 to 2·9
Nickel	8·8	Glass	2·5 „ 3·5
Iron	7·8	Indiarubber	·9
Tin	7·3	Coal-tar	1·01
Zinc	6·9	Earth	1·1 to 1·7
Aluminium	2·7	Granite	2·66
Felspar	2·4 to 2·6	Quicklime	·85
Pyrites from coal	4·1 „ 4·3	Limestone	2·2
Native sulphur	2·07		

Melting Points.

Wrought iron	1,550° Cent.	Aluminium	700° Cent.
Nickel	1,500 „	Zinc	412 „
Steel	1,375 „	Lead	326 „
Cast iron	1,000 to 1,200 „	Tin	230 „
Copper	1,100 „	Mercury	−39 „

English Equivalents of Metric System Units, &c.

1 metre - -	= 39·37 English inches.
1 kilogram (kilo) -	= 2·20 pounds.
1 litre (cub. decimetre)	= 1·76 pints or 61·02 cubic inches.
1 gram - - -	= 15·43 grains.
1 gallon of water weighs 10 lbs. ∴ 224 gallons = 1 ton.	
1 cubic inch - -	= 16·386 cubic centimetres.
1 cubic centimetre	= ·061 cubic inch.
1 oz. avoirdupois -	= 28·35 grams.
1 lb. „ -	= 453·59 „
1 ton - - -	= 1016·0 kilograms.
1 cubic foot - -	= 28,317 cubic centimetres.
1 inch - - -	= 25·4 millimetres.
1 gallon - -	= 4·543 litres.
1 „ - - -	= ·16046 cubic foot or 277·27 cubic inches.
1 cubic foot - -	= 6·23 gallons.
1 gallon per minute	= 9·627 cubic feet per hour.
1 litre - - -	= ·0353 cubic foot.
1 „ - - -	= ·22 gallon.

Weight of 1 Cubic Foot of Material (approx.).

Coal (stored) - -	54 lbs.	Earth - - -	85 lbs.
„ (disintegrated slack)	58 „	Brickwork - - -	87 „
Coke - - - -	24 „	Concrete - - -	92 „
„ breeze - -	45 „	Water - - -	62½ „
Lime - - - -	50 „	Tar - - - -	65 „
Bog ore - - -	54 „	Pitch - - -	70 „

Heat Units.

The British Thermal Unit (B.T.U.) is the quantity of heat required to raise 1 lb. of distilled water 1° Fahr.

The Calorie or French unit of heat, also called the Large Calorie, is the quantity of heat required to raise 1 kilogram of water through 1° Cent.

The Small Calorie is the quantity of heat required to raise 1 gram of water from 0° to 1° Cent.

The Pound-Centigrade Unit is the quantity of heat required to raise 1 lb. water from 0° to 1° Cent.

Consequently 1 British Thermal Unit is equivalent to ·252 large calories, or 252 small calories, or ·555 pound-centigrade units.

Calorific Values of Various Substances calculated from their Chemical Composition.

	B.T.U. per Lb.
Carbon burning to carbon dioxide	14,650
Coal	12,000 to 15,000
Tar	14,000
Coke	13,500
Peat	5,500 to 8,000
Sulphur	4,000
Wood	5,000 to 8,000

1 cubic foot of Ammonium Sulphate weighs approximately 50 lbs.; 1 cubic yard about 12½ cwt.

1 ton of Sulphate requires about 19 cwt. of sulphuric acid for its manufacture.

Conversion of Thermometer Degrees.

° Cent. to ° Fahr. Multiply by 9, divide by 5, add 32.
° Fahr. to ° Cent. Subtract 32, multiply by 5, divide by 9.

Rough Estimation of Temperatures.

Just glowing in the dark	about 525° Cent.
Dark red	„ 700 „
Cherry red	„ 910 „
Bright cherry red	„ 1,000 „
Orange	„ 1,160 „
White	„ 1,300 „
Dazzling bluish white	„ 1,500 „
Electric arc	„ 3,500 „

Proximate Analyses of Coals.

District.	Ash.	Sulphur.	Volatile Matter.	Fixed Carbon.
	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Lancashire	2 to 7	1 to 2·5	32 to 40	50 to 60
Yorkshire	1·5 „ 6	trace to 1·5	30 „ 40	55 „ 65
Derbyshire	1·2 „ 6	·7 to 2·0	35 „ 45	50 „ 60
Welsh (steam)	2 „ 6	·5 „ 1·5	5 „ 12	85 „ 93
	2 „ 7	·7 „ 2·0	30 „ 40	50 „ 60
Scotch	1·2 „ 8	·10 „ 1·5	30 „ 45	50 „ 65
Notts	2·5 „ 8.0	·5 „ 2·0	30 „ 42	50 „ 60
Irish	9·3	1·73	42·4	47·4
Russian	12·0	·59	24·0	63·5
Anthracite	2 to 8	trace to 2·0	7 to 10	85 to 90
Cannel	5 „ 20	1 to 2·5	40	50

Average Composition of Coals from Different Localities.

(*Ultimate Analyses.*)

(Phillips' Admiralty Coal Investigation.)

Locality.	Spec. Gr.	Carbon.	Hydrogen.	Nitrogen.	Sulphur.	Oxygen.	Ash.	Coke.
Average of—	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
36 samples from Wales .	1·315	83·78	4·79	0·98	1·43	4·15	4·91	72·60
18 ,, ,, Newcastle	1·256	82·12	5·31	1·35	1·24	5·69	3·77	60·67
28 ,, ,, Lancashire	1·273	77·90	5·32	1·30	1·44	9·53	4·88	60·22
8 ,, ,, Scotland	1·259	78·53	5·61	1·00	1·11	9·69	4·03	54·22
7 ,, ,, Derbyshire	1·292	79·68	4·94	1·41	1·01	10·28	2·65	59·32

Weights of Gaseous Compounds.

	100 Cub. Feet Weigh in Lbs.		100 Cub. Feet Weigh in Lbs.
Air	7·6	Coal gas	3·83
Ammonia	4·51	Hydrogen	0·53
Carbon dioxide	11·71	Marsh gas	4·3
Carbon monoxide	7·3	Nitrogen	7·44

Approximate Composition of Gaseous Fuels.

	Marsh Gas.	Hydrogen.	Carbon monoxide.	Carbon dioxide.	Nitrogen.	B.T.U. per Cub. Ft
	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	
Blast-furnace gas (coke fed)	...	2·5	28·0	6·0	63·5	120
Producer gas	1·2	13·0	24·6	4·8	56·4	140
Mond gas	1·5	26·0	12·0	15·0	45·5	160
Coke oven gas	35·0	54·0	1·0	1·0	9·0	540
Illuminating gas	36·0	50·0	5·0	0·4	3·5	650

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